Remedial Investigation/ Feasibility Study

Crab Orchard National Wildlife Refuge

U.S. Fish and Wildlife Service
U.S. Department of Interior
Marion, Illinois
and
Sangamo-Weston, Inc.
Atlanta, Georgia

June 1985

O'BRIEN & GERE

O'BRIEN & GERE

June 27, 1985

U.S. Fish and Wildlife Service
1830 Second Avenue
Rock Island, Illinois 61201

Attn: Mr. Richard Ruelle

Re: Remedial Investigation/
Feasibility Study

Crab Orchard National
Wildlife Refuge

File: 3114.001

Gentlemen:

Attached is the Work Plan for the RI/FS to be conducted at the Crab Orchard National Wildlife Refuge. In addition, the following documents are also included as Appendices to the Work Plan:

- Quality Assurance Project Plan
- Site Sampling Plan
- Site Health and Safety Plan.

The Work Plan and Appendices present in detail the protocols to be employed in performing the work as specified in the Scope of Work dated June 1985.

It is currently anticipated that field efforts will begin with the week of July 15. The initial efforts will include the site safety walk-through, geophysical surveys and identification of sampling sites. The bulk of Phase I sampling will be conducted during the months of August and September.

Comments by members of the Primary Contacts group relevant to the field program should be directed to Richard Ruelle by July 12.

Very truly yours,

O'BRIEN & GERE ENGINEERS, INC.

C.B. Murphy Jr.

Cornelius B. Murphy, Jr., Ph.D.
Senior Vice President

SRG:djb

cc: Primary Contact List

3114.001

WORK PLAN

REMEDIAL INVESTIGATION/FEASIBILITY STUDY

CRAB ORCHARD NATIONAL WILDLIFE REFUGE

U.S. FISH AND WILDLIFE SERVICE

U.S. DEPARTMENT OF INTERIOR

MARION, ILLINOIS

AND

SANGAMO-WESTON, INC.

ATLANTA, GEORGIA

O'BRIEN & GERE ENGINEERS, INC.
1304 BUCKLEY ROAD
SYRACUSE, NEW YORK 13221

JUNE 1985

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INTRODUCTION

General

Crab Orchard National Wildlife Refuge (CONWR or the Refuge) is located in southern Illinois primarily within Williamson County, but also extends into neighboring Jackson, Union and Johnson Counties. There are twelve lakes located within the Refuge including Crab Orchard Lake. Crab Orchard Lake was completed in 1940 and has a surface area of 6,965 acres, a maximum depth of 30 feet and 635 acre-feet of storage capacity. The watershed drainage area is 109,261 acres. In addition to supporting an active sport fishing population, the lake serves as water supply (approx. 280,000 gallons per day) for the Refuge and Federal Penitentiary located southeast of the Refuge. The City of Marion also has a supplemental water intake in the Lake which has rarely been used.

The Refuge is administered by the U.S. Fish and Wildlife Service (FWS) of the Department of the Interior (DOI). During the early 1940s and continuing to the present, a number of industries have been active on the Refuge. Industrial activity was especially heavy during World War II when as many as 10,000 persons were employed by a number of defense-related industries.

Background

Previous sampling conducted by FWS and others has shown measurable concentrations of heavy metals and PCBs in fish and other biological species around the Refuge as well as at selected soil and sediment sampling locations. As a result of these previous studies and

from historic information provided by the Refuge Manager, 32 sites and two control areas have been identified for further evaluation. These are listed on Table 1 and located on Figure 1. These sites represent a number of potentially affected matrices including landfills, ponds, surface waters and surface soils as well as Crab Orchard Lake and its sediments and biota. These sites will be evaluated within the context of 13 geographic groupings since many elements of potential impact to receptors will be common to each geographic group. A discussion of background information on each site is presented in Appendix B, the Site Sampling Plan.

This Work Plan has been developed in response to the Scope of Work specified by FWS and Sangamo-Weston, Inc. and dated June 1985. This scope is consistent with U.S. EPA guidance criteria as published in Guidance on Remedial Investigations Under CERCLA (May 1985) and Guidance on Feasibility Studies Under CERCLA (April 1985).

The Work Plan is presented in two parts: 1) Remedial Investigation and 2) Feasibility Study, collectively referred to as RI/FS. In addition to providing manpower and equipment to perform those services specified in the Work Plan, O'Brien & Gere will provide temporary office space on the refuge, as needed, and will be responsible for controlling access to sampling sites during the period of sampling. O'Brien & Gere and any subcontractors used on this project will perform analyses of blank, duplicate and spiked samples as detailed in the Work Plan and Appendix A, the Quality Assurance Project Plan. The Principal Contacts List and a description of responsibilities is included in Appendix A.

The primary objective of the RI/FS is to recommend the most cost-effective source control and off-site remedial actions. Source control remedial actions include measures to prevent, reduce, or eliminate contamination either by containing the hazardous wastes in place or removing them from the site. Off-site remedial actions include measures to mitigate the effects of hazardous waste contamination that has migrated beyond the site. Appropriate source control and off-site remedial actions will be formulated and analyzed in detail after sufficient data have been generated through the remedial investigation.

Based upon existing data, remedial actions that may be appropriate for the CONWR site include, but are not limited to, one or a combination of the following:

- No action.
- Removal and disposal of waste material.
- Solidification or stabilization of waste material.
- In place reconstruction or encapsulation of waste material.
- Continued off-site monitoring.
- Limit access to contaminated areas.
- Groundwater collection and treatment systems.
- Surface water drainage measures to prevent ponding on or near sites of contamination.
- Construction of groundwater barriers.
- Construction of a clay or synthetic cap over contaminated areas.

At the present time, the available data and information on the site are insufficient to allow a definitive selection, screening, and feasibility study of remedial action alternatives without the additional work detailed in the following activities.

At certain points during the RI/FS, submissions will be made that require review and approval. All submissions will be transmitted to FWS who will seek review and approval by U.S. EPA and Illinois EPA (IEPA).

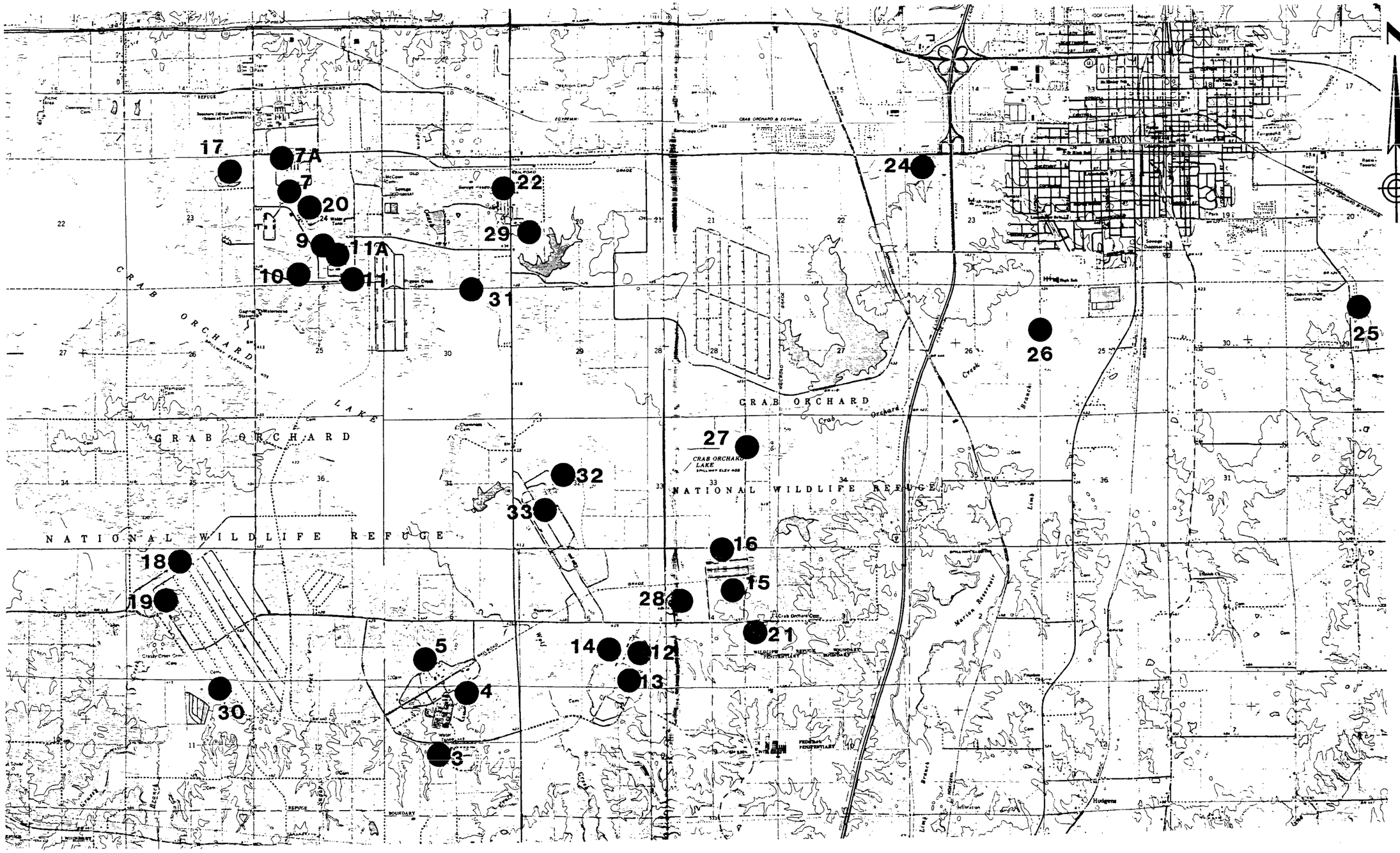
TABLE 1

CRAB ORCHARD REFUGE

SAMPLING SITES

Site #	Type	Name
Group 1		
3	Landfill	Area 11 South Landfill
4	Landfill*	Area 11 North Landfill
5	Pond	Area 11 Acid Pond
Group 2		
7	Surface Water	D Area SE Drainage
7A	Surface Soil	D Area North Lawn
8	Surface Water	D Area SW Drainage
9	Surface Water	P Area NW Drainage
10	Surface Water	Waterworks North Drainage
11	Surface Water	P Area SE Drainage
11A	Surface Soil	P Area North
20	Surface Water	D Area South
Group 3		
12	Landfill*	Area 14 Landfill
13	Surface Soil	Area 14 Change House Site
14	Surface Water	Area 14 Solvent Storage
Group 4		
15	Pond	Area 7 Plating Pond
16	Surface Soil	Area 7 Industrial Site
Group 5		
17	Landfill	Job Corps Landfill
Group 6		
18	Surface Soil	Area 13 Loading Platform
19	Surface Soil	Area 13 Bunker 1-3
30	Control*	Munition Control Site
Group 7		
21	Landfill	Southeast Corner Field
Group 8		
22	Surface Water	Old Refuge Shop
24	Surface Water	Pepsi-West
25	Surface Water	COC at Marion Landfill
26	Surface Water	COC below Marion STP
27	Surface Water	COC below 157 Dredge Area

FIGURE 1



CRAB ORCHARD NATIONAL WILDLIFE REFUGE

LOCATIONS OF SAMPLING SITES

Scale: 1" – 4000'

O'BRIEN & GERE

TABLE 1
(Continued)

CRAB ORCHARD REFUGE

SAMPLING SITES

Site #	Type	Name
Group 9		
28	Landfill	Water Tower Landfill
Group 10		
29	Landfill	Fire Station Landfill
Group 11		
32	Landfill	Area 9 Landfill
33	Surface Soil	Area 9 Building Complex
Group 12		
34	Lake	Crab Orchard Lake
Group 13		
31	Control*	Refuge Control Site

STATEMENT OF WORK

PART I - REMEDIAL INVESTIGATIONS

Purposes

The purposes of this remedial investigation are: 1) to determine the nature and extent of any contaminant problem at several sites (Table 1 and Figure 1) on the Crab Orchard National Wildlife Refuge and tributaries that drain into Crab Orchard Lake and 2) to gather all data necessary to support the Feasibility Study. This will involve the following activities:

- Determine current groundwater gradients.
- Determine the extent of groundwater contamination that has occurred and the rate and direction of contaminant migration.
- Access levels of contaminated soil that may be present adjacent to disposal areas.
- Identify the areal extent of disposed areas.
- Identify specific contaminants which may pose acute or chronic hazards to public health, welfare or the environment.
- Identify pathways of contaminant migration from the sites.
- Define on-site physical features and facilities that could affect contaminant migration, containment, or cleanup.

O'Brien & Gere will furnish all personnel, materials and services necessary for or incidental to performing the remedial investigation on the Crab Orchard National Wildlife Refuge.

Scope

The remedial investigation consists of eight tasks:

Task 1 - Description of Current Situation

Task 2 - Investigation Support

Task 3 - Site Investigation

Task 4 - Preliminary Remedial Technologies

Task 5 - Site Investigations Analyses

Task 6 - Final Report

Task 7 - Community Relations

Task 8 - Additional Requirements

TASK 1 - DESCRIPTION OF CURRENT SITUATION

O'Brien & Gere will describe the background information pertinent to the sites and outline the purpose and need for remedial investigations at those locations. The data gathered during any previous investigations or inspections and other relevant data will be used. A partial list of sources on published and unpublished data available on Crab Orchard Creek watershed and Crab Orchard Lake is included in Attachment 2. This information will be incorporated into Task 6.

A. Site Background

O'Brien & Gere will prepare a summary of the regional location, pertinent area boundary features, surface area, and general site physiography, hydrology, and geology of the sites. The general nature of any contaminant problems, including pertinent history relative to the use of the sites for waste disposal, will be defined.

In addition to telephone contacts with various agencies, one preliminary information-gathering trip will be made to each of the following locations:

1. Illinois Environmental Protection Agency-Springfield;
2. Marion, Illinois to confer with Illinois EPA, and local well drillers;
3. Crab Orchard National Wildlife Refuge to confer with the Fish and Wildlife Service; and
4. Champaign, Illinois to confer with U.S. Geological Survey, and the Soil Conservation Service.

B. Nature and Extent of Problem

The objectives of this task are to collect, review and evaluate all existing information pertinent to the storage, disposal and movement of expected contaminants in the study areas relative to the extent that they affect exposed biological entities. The information will be used to identify the scope of the problem and provide direction to activities carried out in subsequent tasks.

O'Brien & Gere will prepare a summary of the actual and potential on-site and off-site health, and environmental effects, if any, based on current knowledge of the contaminated sites. This may include, but is not limited to the type, physical states, and amounts of the substances involved; the existence and conditions of drums, landfills, and disposal areas; affected media and pathways and exposure;

contaminated releases such as leachate or runoffs; and any human exposure.

Output from this task will take the form of a preliminary hazard assessment. The information will be reviewed to determine the extent and quality of data available on each of the following key factors:

- Receptors: Population of humans and other organisms that may have been or may be exposed to materials originating on-site will be identified.
- Site Characteristics and Pathways: The routes or media by which materials may be escaping from the site will be determined.
- Waste Characteristics: The hazardous properties of the waste including its quantity, chemical form, environmental chemistry and toxicity will be documented.
- Waste Management Practices: The current and past procedures for the storage and prevention of off-site movement of wastes will be reviewed to identify sources, locations and the volume of wastes existing at on and off-site locations.

C. History of Response Actions

This subtask will include the preparation of a summary of previous response actions conducted by either local, State, Federal or private parties, including the site inspections, other technical reports, and their results. The scope of this

RI/FS will address the problems and questions that have been identified during previous work at the sites.

TASK 2 - REMEDIAL INVESTIGATION SUPPORT

Prior to initiating any field investigations, the following preliminary work will be completed.

A. Site Visit

Initial site visits will be conducted to become familiar with site topography, access routes, and proximity of receptors to possible contamination, and collect data to support the Site Health and Safety Plan. Site surveys will be conducted to identify and stake boundaries of known contaminated areas, monitoring wells, and soil borings, and to identify sediment sample locations. A geophysicist will evaluate the applicability of using geophysical methods to determine the existence of contaminant groundwater plumes if necessary. The visit will be used to verify the site information developed in Task 1. The Site Health and Safety Plan will be amended, if necessary, as a result of this visit.

B. Site Maps

O'Brien & Gere will prepare site maps showing all wetlands, water features, drainage patterns, tanks, buildings, utilities, paved areas, easements, right-of-ways, and other features. The site maps and all topographic surveys will be of sufficient detail and accuracy to locate and report all existing and future work performed at the sites. Areas to

be investigated will be mapped using existing topographic maps or aerial photos. After the initial analytical data have been reviewed and where necessary for remedial efforts, the topographic maps will be prepared with 1-foot contours referenced to the National Geodetic Vertical Datum with a scale of 1 inch to 50 feet. The maps will extend 200 feet beyond site boundaries and include all drainages to Crab Orchard Lake.

Boundary lines encompassing contaminanted areas will be identified. The boundary lines for the landfill study sites will be identified using magnetometer and electromagnetic methods. The boundary conditions will be set so that subsequent investigations will cover the contaminated media in sufficient detail to support the feasibility study. The boundary conditions may also be used to identify boundaries for site access control and site security. If necessary, a fence or other security measures may be installed as an initial remedial measure.

C. Dispose of On-Site Generated Waste

All wastes generated by on-site activities will be labelled, drummed and stored within controlled-access areas. Wastes which will be drummed include: all drill cuttings, all purged groundwater from well development, decontamination wash water and disposable protective clothing. These materials, if contaminated, will be properly disposed of during cleanup actions as identified by the feasibility study.

TASK 3 - SITE INVESTIGATIONS

O'Brien & Gere will conduct remedial investigations necessary to characterize the site and its actual or potential hazard to public health and the environment. The site investigations will generate data of adequate technical content to support detailed evaluations of alternatives during the feasibility studies.

The site investigations will be conducted in two phases. Phase I will include geophysical surveys, hydrogeologic investigations, installation of groundwater monitoring wells, and a screening of each site to analyze composited samples for a broad array of potential contaminants as listed in Table 2. Selected samples will be confirmed by a full analysis for the priority pollutants. The screening procedures as documented in the most recent CLP (Contract Laboratory Program for Organic Analysis - Multi-media, Multi-component, Jan. 1985) will be used for the priority pollutants. Actual procedures that will be used for screening as well as full analysis of priority pollutants, dioxins and dibenzo furans are referenced in Appendix A, the Quality Assurance Project Plan. These procedures are consistent with U.S. EPA protocols, or methods specified by the U.S. Fish and Wildlife Service. Strict chain-of-custody procedures will be followed and all sample collection locations or grids will be identified on the site maps established under Task 2.

The sites listed in Table 1 fall under five categories.

1. Landfills
2. Surficial Contaminant Sites
3. Streams
4. Ponds
5. Lake

TABLE 2

RI/FS ANALYTICAL PARAMETERS

1. Purgeable Priority Pollutants
   (Screening and Full Analysis)

2. Acid Extractable Priority Pollutants
   (Screening and Full Analysis)

3. Base/Neutral Extractable Priority
   Pollutants
   (Screening and Full Analysis)

4. Pesticide/PCB Priority Pollutants
   (Screening and Full Analysis)

5. PCB's

6. Metals
   - ICP scan
   - Priority Pollutant Metals
     by AA Spec
   - Mercury

7. Cyanide 40

8. Indicators
   - pH (field)
   - Secific Conductance (field)
   - Total Organic Carbon
   - Total Organic Halogens

9. Explosives Residues by HPLC

10. Nitrogen Series: TKN, NH3N, NO3N

11. PCDD/PCDF
    (Screening and Full Analysis)

12. Cation Exchange Capacity

13. Total Phosphorus

14. Primary and Secondary Drinking Water
    Standards

In addition, two control sites will be included for sampling and analyses. The general rationale employed in developing the sampling and analysis schedules for each category of sites is shown in Table 3. Specific details on the matrix of analytical parameters and sampling and for each site are included in Attachment 1 to Appendix B.

Phase II will consist of additional sampling and analysis to fill in data gaps identified in Phase I and further assess the extent of contamination at each site where materials of concern are found. The general rationale in developing additional sampling and analysis schedules for each category of sites is shown in Table 3.

A. Geophysical Surveys

Geophysical investigations will be conducted to determine the extent of soil and groundwater contamination, if any, in the vicinity of several specified study sites as outlined in Appendix B. In particular, the geophysical investigations will be conducted at areas of suspected landfill activities. investigations will consist of magnetometer and electromagnetic induction (EM) surveys. Initially, test surveys will be conducted to determine the applicability of the method before proceeding with a full scale survey. The instrument will provide surveying capabilities up to a maximum depth of 25 feet. Technical memoranda describing the geophysical investigations with interpretations will be prepared and submitted to the Fish and Wildlife Service before proceeding with the hydrogeologic investigations.

REMEDIAL INVESTIGATION SAMPLING & ANALYSIS SEQUENCE

Site Category	Recon.	Phase I	Phase II	Contingency
Landfills	Geophysics	Cores - depth composites - screening & full priority pollutants & explosives residuals + ICP metals - Install wells-analyze indicators + metals.	Radial & depth cores, and wells for priority pollutants & explosives residuals found in cores & AA metals.	
Surface	Geophysics -locate utilities	Surf. Soils - screening & full priority pollutants and explosive residuals + ICP metals.	Depth soils Radial soils - surf. & depth Runoff - water & sediments & depth profile	
Streams - Waters - Sediments		Upstream/downstream - screening & full priority pollutants & explosive residuals Surf. seds: 2 near shore, 1 near lake - screening & full priority pollutants + expl. + ICP metals	Surf. seds - int + depth seds. - priority pollutants found + AA metals	
Ponds - Waters - Sediments - Groundwater		(Same rationale as streams) (Same rationale as streams) Upgradient/downgradient wells (2) - indicators	Depth profile on sediments priority pollutants + expl. found in waters or seds.	Additional wells
Lake - Waters - Sediments - Biota		5 sites: primary & secondary - Drinking Water stds. (None) Sample & freeze	5 biota sites + 5 use sites: anything found in Phase I 5 sites: parameters found in Phase I parameters found in Phase I	
Control Sites - Lake control - Soil & groundwater control - Clean area - Munitions area		(All analyses included at other sites)	Full scans	

*ICP: Metals analysis by Induced Coupled Plasma Spectrophotometry
AA: Metals analysis by Atomic Adsorption Spectrophotometry

B. Hydrogeologic Investigations

O'Brien & Gere will develop and conduct a program to determine the present and potential extent of groundwater contamination, if any, and evaluate the suitability of the site for on-site waste containment. Efforts will begin with a survey of previous hydrogeologic studies and other existing data (completed as part of Task 1 a and c). The survey will address the degree of hazard, the mobility of chemicals considered, the soil attenuation capacity and mechanisms, discharge/recharge areas, regional flow direction and quality, and effects of any pumping alternative. Subsequent to the survey of existing data, sampling programs will be developed to determine the horizontal and vertical distribution of chemicals considered and predict the long-term disposition of such chemicals. The sampling program will, at a minimum, evaluate factors affecting groundwater performance, background levels of contamination, the type of well construction utilized, the number and location of wells, chain-of-custody, record of samples, and the groundwater sampling method.

A total of sixteen (16) groundwater monitoring wells are specified in Appendix B, the Site Sampling Plan. Three are existing wells at the Area 9 Landfill. The other thirteen proposed monitoring wells will be constructed in accordance with state and local agency regulations. Attempts will be made to procure a driller from within 150 miles of the site. The drilling operation will be conducted under the supervision of O'Brien & Gere personnel.

The monitoring wells will be constructed of 2-inch I.D. Schedule 40, threaded, flush-point PVC pipe with a 5- to 10-foot long, PVC slotted screen or equivalent materials constructed of stainless steel. Each well will be covered with a vented, PVC cap and the pipe will be protected with a 4-inch I.D., locked, protective steel casing, set in a concrete pad. Well screens will be packed with a coarse sand from the bottom of the screen to 6 inches above the screen. Above the course sand pack, a 2-foot bentonite seal (using either granular bentonite or bentonite pellets) will fill the annular space between the soils and the casing. The remaining annular space will be grouted to the ground surface with a tremie pipe to assure that the grout is forced to the bottom and fills all voids. Wells will be developed until no fines are present. If well waters are contaminant-free, in the expert opinion of Federal and State health officials, additional wells will not be installed.

After all wells are installed, the top of the outer, 4-inch casing will be surveyed relative to the National Geodetic Vertical Datum using a benchmark set for the preparation of the site topographic map. The survey record will note the elevation of the top of the inner casing, as well as the distance from the top of the inner casing to the top of the protective casing with the lid open.

Static water levels will be measured in each well by a O'Brien & Gere personnel to the nearest 0.01-foot from the top of the 2-inch well casing. All measurements necessary to

prepare maps of potentiometric surfaces or water tables will be taken on the same day to avoid variation in hydrologic conditions over time. Wells will be allowed to recover at least one week after well installation before static water levels are measured.

C. Sampling and Analyses of Groundwater

Before water sampling begins, the cap will be removed and the well will be monitored immediately for volatile organic compounds. As water is being evacuated, monitoring will continue to assure that proper respiratory protection is being worn as necessary and to identify (qualitatively) contaminated groundwater locations.

Wells will be evacuated prior to sampling. The amount of water evacuated from each well will equal at least five times the amount of standing water in the well casing. Evacuation will continue until no fines are present. If the wells recover slowly and go dry before five well volumes can be purged, a sample will be collected on the following day after recovery. Samples will be obtained with a stainless steel bailer and will be transferred carefully from the bailer to preserved containers and purgeable vials. All wells will be sampled twice during the RI; at least one week after well completion, and again during Phase II sampling. All water evacuated during well development will be drummed and stored onsite until the RI is completed.

All groundwater samples will be collected, preserved, labeled and shipped in accordance with the Quality Assurance Project Plan. Samples will be analyzed for the following parameters in the field:

Temperature

pH

Specific Conductance

Additional monitoring wells will be installed, if necessary after existing on-site wells are sampled and the water analyzed for contaminants of concern. Then, based on the geophysical results (Task 3a) and results of contaminant analyses, the extent and scope of any additional hydrogeologic investigation will be determined.

D. Soil Investigation

O'Brien & Gere will develop and conduct a program to identify the location and extent of surface and subsurface soil, and sediment contamination. This process may overlap with certain aspects of the hydrogeologic study, e.g., characteristics of soil strata are relevant to both the transport of contaminants by groundwater and to the location of contaminants in the soil. These soil samples and an additional number of soil borings will be collected for analysis from various sampling sites around the refuge. The locations where these samples will be collected and sample handling protocols are defined in Appendix B for the various sites suspected of containing contaminants.

E. Surface Water and Sediment Investigation

O'Brien & Gere will develop and conduct a program to determine the extent of water and sediment contamination on selected refuge lakes, marshes, ponds and streams. This process may overlap with the soil investigation; data from lake sediments sampled may be relevant to surface water quality. A survey of existing data on surface water quality and quantity may be a useful first step. The locations where samples will be collected and sample handling protocols are defined in Appendix B.

F. Fish and Wildlife Investigations

Selected species of fish and other aquatic organisms on the refuge will be collected by FWS and analyzed by O'Brien & Gere for residual levels of contaminants previously identified in landfills and other contaminated areas on the refuge. The species, number of organisms to be collected, sampling locations and analytic procedures are identified in Attachment 1, Appendix B, Site 34.

TASK 4 - PRELIMINARY REMEDIAL TECHNOLOGIES

A. Post-Investigation Evaluation

Either during or following the site investigations the O'Brien & Gere will assess the investigation results and recommend preliminary remedial technologies best suited to specific contaminant problems for each site. They will provide the basis for developing detailed alternatives needed for

the completion of the feasibility studies. The data generated during the remedial investigations will generally be limited to accomplish the following:

1. Recommend types of remedial technologies appropriate to physical and site contaminant conditions.
2. Recommending whether or not to remove some or all of the waste for off-site treatment, storage, or disposal.
3. Determine the compatibility of groups of wastes with other wastes and with materials considered as part of potential remedial action. Recommend alternatives for treatment, storage, or disposal for each category of compatible waste.

TASK 5 - SITE INVESTIGATIONS ANALYSIS

The results of Tasks 1 through 4 will be used to prepare a thorough analysis and summary of all site investigations. The objective of this task is to ensure that the investigation data are sufficient in quality and quantity to support the feasibility studies.

The results and data from all site investigations will be organized and presented logically. The geographic groupings listed on Table 1 will form the basic structure for all of the assessments. This will permit the assessment of transport modes and impact to receptors.

A. Data Analysis and Endangerment Assessment

The site investigation data will be analyzed to develop a summary of the type and extent of contamination at the sites. The summary will describe the quantities and concentrations of specific chemicals at each site and ambient levels

surrounding the sites. Ambient samples will be collected from control sites as outlined in Appendix B.

Data collected during the RI phase will also be evaluated to determine if environmental conditions or materials at the site present potential hazards to human health or welfare, or to the environment. Existing standards will be reviewed to help formulate conclusions and recommendations regarding the hazard potential of the site. If additional hazards are identified, the risks associated with each hazard will be summarized.

This analysis will discuss the degree to which either source control or off-site measures are required to significantly eliminate the threat, if any, to public health or the environment. If the results of the investigation indicate that no threat or potential threat exists, a recommendation of no remedial response will be made.

A technical memorandum will be prepared by the Respondents summarizing the hazard evaluation process and presenting the results of the hazard assessment.

TASK 6 - FINAL REPORT

A final RI report will be prepared to consolidate and summarize the data collected during the RI. The report will include a discussion of the data acquired during the RI and the hazard identification and risk potential of the contaminants detected. Ten copies of the remedial investigation report will be submitted to the FWS. The report will be structured to enable the reader to cross-reference with ease.

TASK 7 - COMMUNITY RELATIONS

The Community Relations program is included as Task 7; however, the dissemination of information to the public will be coordinated by the FWS throughout the duration of the study. O'Brien & Gere will provide personnel, at the Service's discretion, to support the programs as community relations must be integrated closely for all remedial response activities.

The objectives of this effort are (1) to keep the community informed as to the study progress, (2) to achieve community understanding of the actions taken, and (3) to obtain community input, and support prior to selection of the remedial alternative(s).

TASK 8 - ADDITIONAL REQUIREMENTS

A. Reporting Requirements

O'Brien & Gere will prepare monthly reports to describe the technical and financial progress of the project. These reports will discuss the following items:

1. Identification of sites on which activity took place and the nature of those activities.
2. Status of work at the site and programs to date.
3. Percentage of completion.
4. Difficulties encountered during the reporting periods.
5. Actions being taken to rectify problems.
6. Activities planned for the next month.
7. Changes in personnel

8. A comparison of target and actual completion dates for each element of activity including project completion and an explanation of any schedule deviations in the work plan.

9. Progress Reports on Items 1 through 8 will be submitted to FWS, who shall in turn relay them to USEPA and IEPA.

10. A Work Plan that includes a detailed technical approach and schedules will be submitted for the proposed feasibility study.

B. Site Health and Safety Plan

Prior to conducting any field activities O'Brien & Gere will provide any necessary modifications to the Site Health and Safety Plan as presented in Appendix C. The plan is consistent with:

Section 111(c)(6) of CERCLA.

EPA Order 1440.3 - Respirator Protection

EPA Order 1440.2 - Health and safety requirements for employees engaged in field activities.

EPA Occupational Health and Safety Manual.

Other EPA guidance as provided.

State Safety and health statutes.

Site conditions.

EPA Interim Standard Operating Safety Guide (September 1982) and applicable OSHA standards.

C. Quality Assurance/Quality Control (QA/QC)

O'Brien & Gere has prepared a Quality Assurance Project Plan (QAPP) for the sampling, analysis, and data handling aspects of the remedial investigation which is presented in Appendix A. The QAPP plan is consistent with U.S. Fish and Wildlife Service, State and Federal EPA requirements. The plan addresses the following points:

1. QA Objectives for Measurement Data, in terms of precision, accuracy, completeness, representativeness and comparability.
2. Sampling Procedures.
3. Sample Custody.
4. Field Equipment, Calibration Procedures, References and Frequency.
5. Internal QC Checks and Frequency.
6. QA Performance Audits, System Audits, and Frequency.
7. QA Reports to Management.
8. Preventative Maintenance Procedures and Schedule.
9. Specific Procedures to be used to routinely assess data precision, representativeness, comparability, accuracy, and completeness of specific measurement parameters involved. This section will be required for all QA project plans.
10. Corrective Action.

D. Site Sampling Plan

A site specific sampling plan has been developed for this Remedial Investigation and is presented as Appendix B. The

sampling plan covers the sampling efforts described in this work plan and addresses the following topics:

- Sample types and tentative locations
- Sample equipment and procedures
- Sample handling, custody procedures, and preservation
- Sample documentation
- Sample shipping
- Analytical arrangements (scheduling)
- Analytical procedures
- QA/QC review procedures of data
- Analytical review of data
- Disposal of unused samples

PART II - FEASIBILITY STUDIES

Purpose

The purposes of the feasibility studies are to develop and evaluate remedial alternatives, and to identify the cost-effective remedial actions to be taken at contaminated sites on the refuge. The following major activities will be accomplished during the feasibility studies:

- Definition and development of alternatives
- Initial screening of alternatives
- Detailed analysis of alternatives
- Selection of the most cost-effective applicable remedial alternative for the site, which will mitigate and minimize damage to and provide adequate protection of public health, welfare, and the environment.
- Preparation of draft and final FS
- Preparation of conceptual design report

O'Brien & Gere will furnish the necessary personnel, materials, and services required to prepare the remedial action feasibility study, except as otherwise specified herein.

Scope

The feasibility study consists of eight tasks numbered 9 through 16:

Task 9 -- Description of Proposed Response

Task 10 -- Development of Alternatives

Task 11 -- Initial Screening of Alternatives

Task 12 -- Laboratory Studies

Task 13 -- Evaluation of the Alternatives

Task 14 -- Final Report

Task 15 -- Conceptual Design

Task 16 -- Additional Requirements

TASK 9 - DESCRIPTION OF CURRENT SITUATION AND PROPOSED RESPONSE

Information on the site background, the nature and extent of the problems and previous response activities presented in Task 1 of the remedial investigation will be incorporated by reference.

Following this summary of the current situation, site specific statements of response purpose, based on the results of remedial investigations, will be presented. The statement of purpose will be organized in terms of components amenable to discrete remedial measures (e.g., a statement of purpose describing the evaluation of alternatives for treatment of any affected groundwater).

TASK 10 - DEVELOPMENT OF ALTERNATIVES

Based on the results of the remedial investigations and consideration of preliminary remedial technologies (Task 4), O'Brien & Gere will develop an appropriate number of alternatives for source control or off-site remedial actions, or both, based on the type and concentrations of contaminants and the physical properties of each site. The number of alternatives may vary for each site; however, a minimum of three will be proposed for each site since the first alternative to consider would be to not relocate the contaminated material (no-action alternative).

A. Establishment of Remedial Response Objectives

This task identifies the site-specific objectives for the response activities. These objectives will be based on threats, if any, to public health and the environment determined through information gathered during the remedial investigation, Section 300.68 of the National Contingency Plan (NCP), USEPA interim guidance, and consultation and with FWS. Preliminary clean-up objectives will be developed in consultation with the FWS and U.S. EPA.

B. Identification of Remedial Alternatives

Develop remedial alternatives for each contaminated site consistent with the, response objectives, and other appropriate considerations. In general, these alternatives will incorporate the following technologies:

- Avoid Technologies: remove the receptor from the affected source. For example, replacement of a contaminated water supply with a new source accomplishes this goal.
- Containment Technologies: prevent or control the movement of contaminant materials. Landfill caps, slurry walls, and drainage controls accomplish this objective.
- Remove Technologies: physically remove contaminants from the area of concern. Excavation and flushing are examples.
- Treatment Technologies: remove contaminants from the affected matrix or convert them chemically to a less hazardous form.

For each of the feasible technologies being evaluated, a list of site characteristics (site area, climate, etc.) will be compiled. This will define the information that must be obtained from the RI, and assist in subsequent decision-making regarding the different alternatives.

A matrix containing the above information will be developed to facilitate compilation of data as they are received. This matrix will also provide a convenient means of presenting data and serve as an aid in the decision-making process of the alternative evaluation. As data from the RI are received and compiled in the matrix, it will become evident if more information is required to properly analyze alternatives. Since this process will occur during the RI, any changes in data gathering requirements will be easily accomplished. Any additional work which becomes identified and is not part of this work plan will be described in a separate work plan and submitted to FWS for review.

Alternatives will include non-clean up (e.g., relocation) and no-action options. These alternatives will be developed in consultation with the FWS.

TASK 11 - INITIAL SCREENING OF ALTERNATIVES

The alternatives developed in Task 10 will be screened by O'Brien & Gere and FWS to eliminate alternatives that are clearly not feasible or appropriate, prior to undertaking detailed evaluations of the remaining alternatives. Screening will be completed within 60 days after identification of the alternatives.

A. Develop Preliminary Screening Criteria

The criteria which will be used in the initial screening of alternatives will be developed by O'Brien & Gere according to currently available guidelines established by the EPA. It is a three-stage screening process which incorporates the components of environmental, engineering and economic criteria.

Environmental Screening Criteria

The response alternatives will be developed to address the following environmental screening criteria:

1. Adverse impacts due to the alternative's implementation that could affect public health or the environment;
2. The effectiveness of the alternative in providing adequate protection of the environment and the public from the hazards posed by the site;
3. The public acceptability of the alternative; and
4. Legal issues which may affect implementation of the alternative.

Engineering Screening Criteria

The response alternatives must meet the following engineering criteria:

1. The alternative must be compatible with all site specific characteristics;
2. The alternative must be compatible with all waste specific characteristics; and

3. The alternative must complete the purpose that it was designed to do in an efficient and reliable manner.

Economic Screening Criteria

The initial economic assessment is intended to provide only an "order of magnitude" cost. The data utilized in the costing must be easy to obtain and need only be accurate within -50% and +100%. Once obtained, these data will be used to estimate alternative costs (including both capital and operation and maintenance costs) and will be presented on a present-worth basis. These costs form the criteria against which the alternatives can be compared.

Criteria Summaries

The criteria established as a result of this subtask will be summarized and arranged into a matrix. This matrix will be submitted to FWS for review prior to proceeding to the initial screening subtask.

B. Initial Screening

Once the criteria are established, the actual initial screening will be performed. The individual or combination of individual remedial technologies will be evaluated utilizing the criteria generated as described above. The evaluation will be conducted using prudent engineering judgment and following the methods in the EPA "Procedural Manual for Screening and

Development of Alternatives". This will reduce the number of alternatives to between three and ten different response alternatives.

TASK 12 - LABORATORY STUDIES (if required)

O'Brien & Gere will conduct any necessary laboratory and bench scale treatability studies required to evaluate the effectiveness of remedial technologies and establish engineering criteria (e.g., leachate treatment, groundwater treatment; compatability of waste/leachate with site barrier walls, cover, and other materials proposed for use in the remedy).

This subtask provides for the performance of additional engineering investigations which may be required to complete the detailed analyses of response alternatives with site and waste specific conditions. These additional investigations will serve to formulate the engineering details for each alternative which will be used in the subsequent assessment and ranking of the alternatives.

The engineering investigations performed during this work task may include, but are not limited to, the following:

- Determination of the physical and/or chemical properties of soils by laboratory analysis and identification of such parameters as transmissivity, void ratio, attenuation capabilities, soil cohesion, and any other necessary physical/ chemical properties;
- A literature review to evaluate compatibility of construction materials and the on-site chemical contaminants, including an investigation of the chemical effects on wells and well

construction materials, materials utilized in treatment systems, pipelines, cover or capping materials, and other construction materials; and

- The examination of Task 3 hydrogeological information, and, if necessary, the development of an aquifer performance test including long-term pump tests. This effort would include the methods of pumping the aquifer, disposition of the pumped water, and temporary treatment technologies.

A. Analysis of Treatability Methods

For any alternative that involves the treatment of contaminated groundwater or removed waste, an analysis may be necessary to determine if the method can effectively treat the groundwater (or waste) to an acceptable level, as determined by regulatory standards. These studies may include a search of published technical literature. Treatability studies will be conducted if necessary to evaluate the technical or economic feasibility of treatment or containment technologies.

It is expected that the scope of this task will depend on the results of Tasks 10 and 11 and therefore will not be complete at the start of Task 13. A separate work plan for any proposed laboratory studies will be submitted to FWS for approval. This submittal will be made in the time frame required to maintain steady progress of the overall feasibility study. (Additional studies also may be conducted during the design phase if needed to refine treatability results or develop detailed design criteria.)

TASK 13 - EVALUATION OF THE ALTERNATIVES

O'Brien & Gere will evaluate the alternative remedies that pass through the initial screening in Task 11 and recommend the most cost effective alternative to the FWS.

A. Detailed Development of Remaining Alternatives

Alternatives developed must meet the applicable requirements of all environmental statutes. The detailed development of the remaining feasible remedial alternatives will include the following element:

1. Description of appropriate treatment and disposal technologies, including:.
   - ° Basic component diagrams for each alternative to be considered, including criteria, quantities of materials to be handled, efficiency of contaminant removal, and other basic information.
   - ° Major equipment needs and utility requirements.
   - ° Conceptual site layout drawings.
2. Special engineering considerations required to implement the alternative (e.g., pilot treatment facility, additional studies needed to proceed with final remedial design).
3. Environmental impacts and proposed methods, and costs, for mitigating any adverse effects.
4. Operation, maintenance, and monitoring requirements of the remedy.
5. Off-site disposal needs and transportation plans.
6. Temporary storage requirements.

7. Safety requirements for remedial implementation (including both on-site and off-site health and safety considerations).

8. A description of how the alternatives could be phased into individual operable units. The description should include a discussion of how various operable units of the total remedy could be implemented individually or in groups, resulting in a significant improvement to the environment or savings in costs.

9. A description of how the alternative could be segmented into areas to allow implementation of differing phases of the alternative.

10. A review of any disposal facilities to ensure compliance with applicable requirements and other environmental laws.

B. Environmental Assessment

The environmental assessment will include investigations will respect to:

1. The adverse environmental impacts (such as noise, air emissions, surface or groundwater discharges, visual impact, dust control, traffic considerations, etc.) of implementation of the alternative;

2. The adequacy of the alternative in remediating the threat to the public health and environment;

3. Public acceptability; and

4. Possible regulatory constraints such as environmental permitting and all Federal, State and local regulations pertaining to the environment.

In addition, each alternative will be assessed in terms of the extent to which it mitigates long-term exposure to any residual chemical substance and protects public health both during and after completion of the remedial action. The assessment will describe the levels and characteristics of chemical substances potential exposure routes, and threat to wildlife and fish population (the endangerment assessment prepared in Task 5a will be used for this). The effect of "no action" will be described in terms of the short term effects, the long term exposure to chemical substances, and resulting public health impacts. Each remedial action recommendation will be evaluated to determine the level of exposure and the reduction over time of such substances. The relative reduction in public health impacts for each alternative will be compared to the no action level. For off-site measures the relative reduction in impact will be determined by comparing residual levels of each alternative with existing criteria, standards or guidelines acceptable to U.S. EPA. For source control measures, or when criteria, standards, or guidelines are not available, the comparison will be made based on the relative effectiveness.

The relative reduction in public health impacts for each alternative will be compared by listing alternatives according to increasing levels of protection. The no action alternative

will serve as the baseline for the analyses. The comparative assessment will include the following elements:

- The adverse environmental impacts of the alternatives, including potential risks to the public during construction and operation.
- The effectiveness in mitigating adverse impacts.
- The adequacy of source control remedial measures.
- The effectiveness of off-site control measures in mitigating the danger or threat of danger to the public or the environment.
- The public acceptability of the alternatives.
- An assessment of the environmental and health risks associated with each alternative.
- The regulatory constraints (environmental permits) that could affect the implementation of the alternatives.

C. Cost Analyses

Evaluate the cost of each feasible remedial action alternative (and for each phase or segment of the alternative). The cost will be presented in the form of present worth and will include the total cost of implementing the alternative as follows:

- Capital Costs;
- Construction Costs;
- Operations and Maintenance Costs; and
- Effects of Health and Safety Requirements on Construction and O&M Costs.

Each cost estimate will be compared following performance of a present worth analysis to consider effects of the time value of money. The ammortization rate used will be selected using US EPA guidance and as agreed upon by FWS.

Sensitivity Analysis

An analysis will be conducted to assess the effect of variations in the parameters used in the preliminary conceptual design of the alternatives. This analysis will determine how the estimated costs are affected by small changes in key parameters. The method used for conducting this analysis is outlined in the "Remedial Action Costing Procedures Manual", developed for the US EPA in 1984.

D. Evaluation and Recommendation of Cost-Effective Alternative

Alternatives will be evaluated using technical, environmental, and economic criteria. At a minimum, the following areas will be used to evaluate alternatives:

1. Reliability. Alternatives that minimize or eliminate the potential for release of waste constituents into the environment will be considered more reliable than other alternatives. For example, disposal methods that would permanently eliminate the potential for the wastes to be recycled back into the environment would be considered more reliable than some other disposal methods. Institutional concerns such as management requirements also will be considered.

2. Implementability. The requirements of implementing the alternatives will be considered, including phasing alternatives into operable units and segmenting alternatives. The requirements for permits, zoning restrictions, right-of-ways and public acceptance are factors to be considered.

3. Operation and Maintenance Requirements. Preference will be given to projects with lower operation and maintenance requirements, other factors being equal.

4. Environmental Effects. Alternatives posing the least impact (or greatest improvement) on the environment will be favored.

5. Safety Requirements. On-site and off-site safety requirements during implementation of the alternatives should be considered. The safest alternatives with the lowest cost will be favored.

6. Cost. The remedial alternative with the lowest total present worth cost will be favored. Total present worth cost includes capital cost of implementing the alternative and cost of operation and maintenance of the proposed alternative.

An assessment summary identifying each alternative with regard to the aspects listed above will be prepared. The assessments conducted above will be compiled and ranked. Procedures set forth in the "Superfund Feasibility Study

Guidance Document" will be followed. The ranking process will consist of the following work items:

1. Group Specific Ranking. Each alternative will first be ranked within the individual groups of economic, environmental, and engineering feasibility. This will provide a clear identification of strengths and weaknesses of each alternative within a specific category.
2. Overall Ranking. With the above information, the alternatives will be ranked using all three groups. This ranking will utilize all of the input from FWS, State, Federal and local agencies; input from the public received during the feasibility study, from information generated during the assessments. The ranking will be based on sound engineering judgment.

O'Brien & Gere will recommend the most cost-effective alternative that provides an acceptable level of risk to potential receptors. The recommendation will be justified by stating the relative advantages over other alternatives considered. All alternatives shall be given equal consideration. The lowest cost alternative that is technologically feasible and reliable and provides adequate protection of public health, welfare, or the environment will be considered the best cost-effective alternative.

During this task, US EPA and FWS may determine that additional data are necessary to be obtained or engineering evaluations are needed to be conducted in addition to

previously approved reports or documents in order to properly conduct the comparative ranking of the alternative being considered. If this is required, a separate work plan will be submitted to FWS and the schedule shall be adjusted accordingly.

E. Preliminary Report

O'Brien & Gere will prepare a preliminary report presenting the results of Tasks 9 through 13 and the recommended remedial alternatives. Ten copies of the preliminary report will be submitted to the FWS.

After alternatives have been evaluated and the most cost-effective and environmentally sound alternative has been selected, a public hearing will be held after two weeks notice has been given. Written comments from government agencies and individuals on the alternatives will be accepted for three weeks following the hearing.

TASK 14 - FINAL REPORT

Following the public comment periods, a final report will be submitted to the FWS. The report will include the results of Tasks 9 through 14. Ten copies will be submitted to the FWS. The Service will review the final report, discuss alternatives with O'Brien & Gere, Sangamo-Weston and cooperating agencies and select a remedial alternative.

TASK 15 - CONCEPTUAL DESIGN

The following conceptual design elements will be developed by O'Brien & Gere as required for the remedial actions selected.

- A conceptual plan view drawing of the overall site, showing general locations for project actions and facilities.
- Conceptual layouts (plan and cross sectional views where required) for the individual facilities, other items to be installed, or actions to be implemented.
- Conceptual design criteria and rationale.
- A description of types of equipment required, including approximate capacity, size, and materials of construction.
- Process flow sheets, including chemical consumption estimates and a description of the process.
- An operational description of process units or other facilities.
- A description of unique structural concepts for facilities.
- A description of operation and maintenance requirements.
- A discussion of potential construction problems.
- Right-of-way requirements.
- A description of technical requirements for environmental mitigation measures.
- Additional engineering data require to proceed with design.
- Construction permit requirements.
- Implementation cost estimate.
- Annual O&M cost estimates.
- Preliminary project schedule.

TASK 16 - ADDITIONAL REQUIREMENTS

The same reporting requirements which are identified in Task 8 of the Remedial Investigation scope of work will be met during the Feasibility Study.

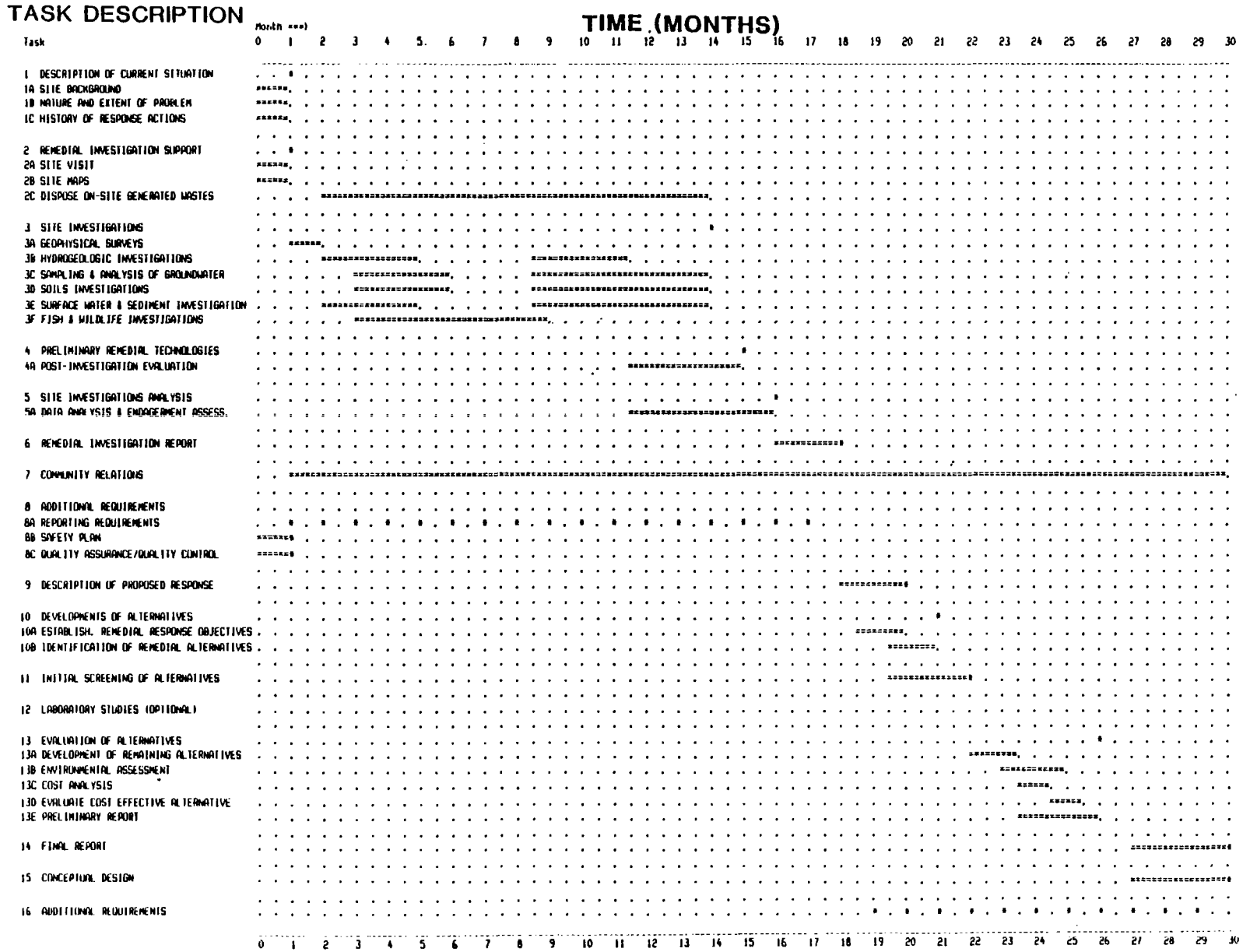
PROJECT SCHEDULE

The proposed schedule for implementing the work detailed in this Work Plan and associated appendices is illustrated on the attached Figure. This is a preliminary schedule developed for planning purposes. Several tasks identified in the Work Plan (i.e., Tasks 3, 12 and 13) emphasize uncertainties or contingent items which may be defined at a later date, depending on the results of analytical data or engineering assessments. The proposed schedule attempts to accommodate these uncertainties; however, schedule modifications may be necessary as these tasks are encountered.

In addition, a table of deliverables is also attached. This table identifies key periods at which materials will be transmitted and discussed with FWS.

CRAB ORCHARD NATIONAL WILDLIFE REFUGE

PROJECT SCHEDULE



CRAB ORCHARD NATIONAL WILDLIFE REFUGE
PROJECT DELIVERABLE DATES

TASK No.	TASK	OUTPUT	DELIVARABLE DATE
1	DESCRIPTION OF CURRENT SITUATION	. Work Plan	Jun. 28, 1985 *
		. Draft Final Report Section	Jul. 31, 1985
1A	SITE BACKGROUND		
1B	NATURE AND EXTENT OF PROBLEM		
1C	HISTORY OF RESPONSE ACTIONS		
2	REMEDIAL INVESTIGATION SUPPORT	. Draft Final Report Section	Jul. 31, 1985
2A	SITE VISIT		
2B	SITE MAPS		
2C	DISPOSE ON-SITE GENERATED WASTES		
3	SITE INVESTIGATIONS	. Investigations,	Aug. 31, 1986 *
3A	GEOPHYSICAL SURVEYS	Draft Final Report Section	
3B	HYDROGEOLOGIC INVESTIGATIONS		
3C	SAMPLING & ANALYSIS OF GROUNDWATER		
3D	SOILS INVESTIGATIONS		
3E	SURFACE WATER & SEDIMENT INVESTIGATION		
3F	FISH & WILDLIFE INVESTIGATIONS		
4	PRELIMINARY REMEDIAL TECHNOLOGIES	. Draft Final Report Section	Sep. 30, 1986
4A	POST-INVESTIGATION EVALUATION		
5	SITE INVESTIGATIONS ANALYSIS	. Draft Final Report Section	Oct. 31, 1986
5A	DATA ANALYSIS & ENDAGERMENT ASSESS.		
6	REMEDIAL INVESTIGATION REPORT	. Final Reports	Dec. 31, 1986 *
7	COMMUNITY RELATIONS		
8	ADDITIONAL REQUIREMENTS		
8A	REPORTING REQUIREMENTS	. Monthly Progress Reports	First Day of Month
8B	SITE HEALTH & SAFETY PLAN		Jun. 28, 1985
8C	QUALITY ASSURANCE PROJECT PLAN		Jun. 28, 1985
9	DESCRIPTION OF PROPOSED RESPONSE	. Draft Final Report Section	Feb. 28, 1987
10	DEVELOPMENTS OF ALTERNATIVES	. Preliminary Alternatives,	Mar. 31, 1987
10A	ESTABLISH. REMEDIAL RESPONSE OBJECTIVES	Draft Final Report Section	
10B	IDENTIFICATION OF REMEDIAL ALTERNATIVES		
11	INITIAL SCREENING OF ALTERNATIVES	. Draft Final Report Section	Apr. 30, 1987 *
12	LABORATORY STUDIES (OPTIONAL)		
13	EVALUATION OF ALTERNATIVES	. Draft Feasibility Report	Aug. 31, 1987 *
13A	DEVELOPMENT OF REMAINING ALTERNATIVES		
13B	ENVIRONMENTAL ASSESSMENT		
13C	COST ANALYSIS		
13D	EVALUATE COST EFFECTIVE ALTERNATIVE		
13E	PRELIMINARY REPORT		
14	FINAL REPORT	. Final Reports	Dec. 31, 1987 *
15	CONCEPTUAL DESIGN	. Final Reports	Dec. 31, 1987 *
16	ADDITIONAL REQUIREMENTS		

ATTACHMENT 1

PRIMARY CONTACTS

PRIMARY CONTACTS

Name and Responsibility	Organization and Address	Phone Number
Dr. James Elder Regional Resource Contaminants Assessment Coordinator	U.S. Fish and Wildlife Service Federal Building, Fort Snelling Twin Cities, MN 55111	612/725-3536
Mr. Wayne Adams Refuge Manager	U.S. Fish and Wildlife Service Crab Orchard National Wildlife Refuge P.O. Box J Carterville, IL 62918	618/997-3344
Dr. Dave Stallings Dr. Jim Petty Quality Control/ Quality Assurance	Columbia National Fisheries Research Laboratory U.S. Fish and Wildlife Service Route 1 Columbia, MO 65201	314/875-5399
Mr. Dick Ruelle Illinois Resource Contaminants Assessment Coordinator	U.S. Fish and Wildlife Service 1830 Second Avenue Rock Island, IL 61201	309/793-5800
Contracting and General Services	U.S. Fish and Wildlife Service Federal Building, Fort Snelling Twin Cities, MN 55111	612/725-3580
Mr. Rodney Gaither On-Scene Coordinator	U.S. Environmental Protection Agency 230 South Dearborn Street Chicago, IL 64604	312/886-4735
Mr. Bob Cowles Superfund Coordinator	Illinois Environmental Protection Agency 2200 Churchill Road Springfield, IL 62706	217/782-6760
Mr. Joe Stuart Illinois EPA Representative	Illinois Environmental Protection Agency 2209 West Main Marion, IL 62959	618/997-4371
Mr. Mike Carter Illinois Dept. of Conservation Representative	Regional Fish & Wildlife Manager Illinois Dept. of Conservation R.R. 4, Box 68 Benton, IL 62812	Office: 618/435-8138 Home: 618/883-5961

Ms. Vanessa Musgrave Community Relations	U.S. Environmental Protection Agency 230 South Dearborn Street Chicago, IL 64604	312/886-6128
Mr. Jim Ross Community Relations	U.S. Fish and Wildlife Service Federal Building, Fort Snelling Twin Cities, MN 55111	612/725-3519
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Mr. Les Frankland Illinois Dept. of Conservation	Illinois Dept. of Conservation 424 Lincoln Tower Plaza Springfield, IL 62706	217/782-6424
Ms. Carol B. Luly Community Relations	Illinois Environmental Protection Agency 2009 Mall Street Collinsville, IL 62234	618/345-6220
Ms. Alison Ling Office of Soliciter U.S. Department of Interior	U.S. Department of the Interior Room 4354 18th & C Streets, N.W. Washington, D.C. 20240	202/343-1301
Mr. David M. Taliaferro Attorney, U.S. EPA	U.S. Environmental Protection Agency 230 South Dearborn Street Chicago, IL 64604	312/886-6826
Dr. Cornelius B. Murphy, Jr. O'Brien & Gere	O'Brien & Gere Engineers, Inc. P.O. Box 4873 1304 Buckley Road Syracuse, NY 13221	315/451-4700
Mr. John Hanson Beveridge & Diamond	Beveridge & Diamond, P.C. 1333 New Hampshire Ave., N.W. Washington, D.C. 20036	202/828-0285
Mr. Christian E. Liipfert Sangamo Weston, Inc.	Sangamo Weston, Inc. P.O. Box 48400 Atlanta, GA 30362	404/449-9006

ATTACHMENT 2

Partial List of Published and Unpublished Data Available on Crab Orchard Creek Watershed and Crab Orchard Lake.

TABLE 1. Partial summary of monitoring data and information available on Crab Orchard Creek Watershed and Crab Orchard Lake. Compiled by R.L. Hite, IEPA Monitoring Unit, Marion.

TYPE OF INFORMATION OR PUBLICATION TITLE	GENERAL AREA	DESCRIPTION OF DATA	DATE(S) OR PERIOD OF RECORD	AVAILABILITY
Water and Land Resources of the Crab Orchard Lake Basin. J. B. Stall, et al, 1958.	Crab Orchard Lake and Watershed	Historical, sedimentation, sediment-soil characteristics, water supply information.	1954	Published by State Water Survey
Big Muddy River Comprehensive Basin Study, Vols 1-7. Prepared by several agencies under the supervision of Big Muddy River Basin Coordinating Committee	Big Muddy Basin including Crab Orchard Creek Basin	Climatology, meteorology, surface water hydrology, ground water availability, flood control & drainage, agriculture and economics.	1968 - 1971	Library
Water Quality Management Basin Plan for the Big Muddy River Basin. IEPA, 1976.	Big Muddy Basin including Crab Orchard Creek Basin IEPA segment A-05, 06, 07	General water quality data, water quality violations, point source information (pages IV-87 to IV-96).	1975 Cal year	IEPA
City of Marion Report responding to Pollution Control Board Order (PCB 75-220)	Crab Orchard Lake	Phytoplankton Survey	June 24, 1975 July 8, 1975	IEPA Monitoring Unit-Marion
Biological Investigation of the Crab Orchard Creek Basin, Summer 1975. R.L. Hite and M. King	Crab Orchard Creek and Crab Orchard Lake	Water Quality Data Biological Data on Basin Streams, Sediment Chemistry Basin Streams & Lakes; Fish Flesh Data Crab Orchard Lake	1975-1976 W.Q. Data for 1972-1976	IEPA
Joint Water Quality Study of Crab Orchard Lake and Bathing Beaches. Hewitt, J. 1976	Crab Orchard Lake	Bacteriological Data	1976	IDPH-Marion
Report on Crab Orchard Lake. EPA Region V, Working paper No. 301 National Eutrophication Survey	Crab Orchard Lake	Water Quality Data, point source loadings	1973-1974	Region V EPA or IEPA
IEPA, Ambient Water Quality Data	Station MD-04; Crab Orchard Creek E. of Marion at Route 13	Routine Water Quality Parameters, Nutrients, Metals, etc.	1977-1980	STORET, IEPA, IOSN reports, and USGS Water Resources Data Series
IEPA Ambient Water Quality Data	Station MD-03; Crab Orchard Creek at I-57, 1.5 miles SE of Marion	Routine Water Quality Parameters, Nutrients, Metals, etc.	1972-1977	STORET-IEPA; USGS Water Resource Investigations 79-25 (1979)

Table 1 (Con't).

TYPE OF INFORMATION OR PUBLICATION TITLE	GENERAL AREA	DESCRIPTION OF DATA	DATA(S) OR PERIOD OF RECORD	AVAILABILITY
IEPA Ambient Lake Monitoring Program Data	Marion City Reservoir; (Lake Code RM-A05-L)	Water Quality Data	1977; 1979;1981-1983	STORET-IEPA Monitoring Unit-Springfield
IEPA Ambient Lake Monitoring Program Data	Devils Kitchen Lake (Lake Code RM-A06-J)	Water Quality Data	1977; 1979; 1982-1984	STORET-IEPA Monitoring Unit-Springfield
IEPA Ambient Lake Monitoring Program Data	Little Grassy Lake (Lake Code RM-A06-R)	Water Quality Data	1977; 1981	STORET-IEPA Monitoring Unit-Springfield
Illinois Water Quality Inventory Report 1980-1981 Volume II; IEPA Monitoring Unit Report (305b)	Statewide, Data on Marion Reservoir, Crab Orchard Lake	Includes statistical summary of parameter values	1979-1981	Monitoring Unit Marion or Springfield
Classification/Needs Assessment of Illinois Lake for protection, restoration, and management. IEPA Monitoring Unit Report, D.F. Sefton and J.R. Little	State Lakes; includes Crab Orchard Lake, Marion City Reservoir, Devils Kitchen and Little Grassy	Water Quality Ratings, improvement potential ratings, public benefits rating and overall lake calssification	Published February 1984	IEPA Monitoring Unit-Springfield
Illinois Water Quality Inventory Report 1982-1983 Volume II; IEPA Monitorial Unit Report 305b	Statewide,Crab Orchard	Whole fish data for Crab Orchard	March 3, 1983	STORET-IEPA Monitoring Unit-Springfield
Fish Contaminant Data	7 samples from RT 148 area; 14 samples from dam area	PCB concentrations in fish fillets	March 1983	IDOC
Survey for Polychlorinated Biphenyls on Crab Orchard National Wildlife Refuge. USF & W Report, R. Ruelle	Crab Orchard Refuge and Lake	PCB data in dump soils, lake & tributary sediments, lake fish (whole fish)	June-July 1982	USF & W - Ruelle
Mercury Levels in Crab Orchard Lake Largemouth Bass 1982 USF & W Report. R. Ruelle	Crab Orchard Lake	Mercury Concentrations in fish fillets	June 1982	USF & W - Ruelle
Survey for Lead on Crab Orchard National Wildlife Refuge. USF & W Report. R. Ruelle	Crab Orchard National Refuge And Lake	Lead concentrations in soils, earthworms, mammals, and Crab Orchard whole fish		
University of Nebraska Lab results for Dump Soils	Sangamo Dump	Tetra and Pentachlorodibenzodioxins	August 26, 1983	USF & W - Ruelle
Fish Contaminant data	Marion City Reservoir	Mercury, PCB's and other organochlorine compound in fish fillets	May 7, 1984	IDOC
Fish Contaminant Data	Crab Orchard Lake East of Highway 148 and near Dam	Mercury, PCB's and other organochlorine compounds in fish fillets	March 14, 1983 & May 18, 1984	IDOC
IEAP Draft Report Polychlorinated Biphenyl Monitoring; Crab Orchard Lake, 1983-1984	13 Crab Orchard Lake Sites; one site in Crab Orchard Creek	Water chemistry including organics/metals and routine ambient parameters; sediment chemistry	May 24, 1983 May 21, 1984	IEPA Monitoring Unit-Springfield

Table 1 (Con't)

TYPE OF INFORMATION OR PUBLICATION TITLE	GENERAL AREA	DESCRIPTION OF DATA	DATE(S) OR PERIOD OF RECORD	AVAILABILITY
IEPA Ambient Water Quality Data	Station MD-02; Crab Orchard Creek downstream from Crab Orchard Lake Dam	Routine Water Quality Parameters, Nutrients, Metals, etc.	1972-1984	STORET, IEPA 305b reports, and USGS Water Resource Data Series
Biological and Water Quality Survey of Crab Orchard Creek in the vicinity of Marion, IEPA Monitoring Unit Report M. Kelly et. al.	Crab Orchard Creek	Macroinvertebrate, Water Quality dial, oxygen	1979-1980	IEPA Monitoring Unit-Marion
IEPA Municipal Wastewater Treatment Facility Data	City of Marion, Carterville, Crab Orchard Refuge, Pittsburg, Marion Federal Penitentiary	Effluent Data-facility information	1972-1984	IEPA Field Operations Section (DWPC) Marion
Limnology of Devils Kitchen Lake IEPA Monitoring Unit Report R.L. Mile et.al.	Devils Kitchen Lake and Grassy Creek	Water Quality Data for Devils Kitchen Lake and Grassy Creek Stream Macroinvertebrates, Lake Sediment Chemistry	1979	IEPA-Monitoring Unit-Springfield
Limnology of 63 Illinois Lakes. IEPA Monitoring Unit Report, Sefton et.al.	State Lakes including data from Crab Orchard Lake	Water Chemistry, phytoplankton sediment chemistry	1979	IEPA Monitoring Unit-Springfield
Chemical Analysis of Surficial Sediments from 63 Illinois Lakes IEPA Monitoring Unit Report, M. Kelly and R.L.Mile	State Lakes, Crab Orchard Lake, Marion City Reservoir, Devils Kitchen	Sediment Chemistry	1979	IEPA Monitoring Unit-Springfield
Evaluation of Illinois Stream Sediment Data 1974-1980; IEPA Monitoring Unit Report	Illinois Streams including Crab Orchard Creek and other sites in watershed	Sediment Chemistry Data, Classification of Stream Sediments	1974-1980	IEPA Monitoring Unit-Marion
Assessment and Classification of Illinois Lakes, Volume I, IEPA Report, D. Sefton	State Lakes including Crab Orchard Lake	Lake Morphometry, physical data, inventory of problems, phytoplankton	1978	IEPA Monitoring Unit-Springfield
Assessment and Classification of Illinois Lakes, Volume II IEPA Report, D. Sefton	State Lakes including Crab Orchard Lake	Physical Data, Water Quality Data	1977	IEPA Monitoring Unit-Springfield
IEPA Ambient Lake Monitoring Program Monitoring Data	Crab Orchard Lake four Sites; (Lake Code RM-AD6-A)	Water Quality Data	1977; 1979-1984	STORET-IEPA Monitoring Unit-Springfield

Work Plan Appendix A
Quality Assurance Project Plan

Remedial Investigation/ Feasibility Study

Crab Orchard National Wildlife Refuge

U.S. Fish and Wildlife Service
U.S. Department of Interior
Marion, Illinois
and
Sangamo-Weston, Inc.
Atlanta, Georgia

June 1985

APPENDIX A

QUALITY ASSURANCE PROJECT PLAN

REMEDIAL INVESTIGATION/FEASIBILITY STUDY

CRAB ORCHARD NATIONAL WILDLIFE REFUGE

U.S. FISH AND WILDLIFE SERVICE

U.S. DEPARTMENT OF INTERIOR

MARION, ILLINOIS

AND

SANGAMO-WESTON, INC.

ATLANTA, GEORGIA

O'BRIEN & GERE ENGINEERS, INC.
1304 BUCKLEY ROAD
SYRACUSE, NEW YORK 13221

JUNE 1985

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INTRODUCTION

Each investigator generating data has the responsibility to implement minimum procedures to assure that the precision, accuracy, completeness, and representativeness of the data are known and documented. In addition, the investigator should specify the quality levels that data must meet in order to be acceptable. To ensure that this responsibility is met uniformly, each investigator must have a written QA Project Plan (QAPP) covering each project that is investigated.

This QA Project Plan has been prepared by O'Brien & Gere Engineers for the Crab Orchard National Wildlife Refuge Site. It is in the format specified in EPA document QAMS-005/80 entitled "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans." The QAPP presents, in specific terms, the policies, organization, objectives, functional activities, and specific QA and quality control (QC) activities designed to achieve the data quality goals of the specific project. Where possible, existing QA/QC guidelines, policies, programs, etc., are incorporated into the QAPP by reference.

PROJECT DESCRIPTION

The remedial investigation/feasibility study (RI/FS) for the Crab Orchard National Wildlife Refuge Site is intended to determine the nature and extent of contamination, to develop and evaluate remedial alternatives and to identify cost-effective remedial actions to be taken at contaminated sites on the refuge which reduce risks to acceptable levels. To accomplish this, the following tasks will be completed:

- characterize the on-site soil, sediment, water and biological samples for the presence of hazardous contaminants (includes landfill, surface soil, pond and lake water).
- identify pathways of chemical migration from the site.
- characterize the off-site soil, sediment, water and biological samples for key hazardous components.
- determine and describe on-site physical features that could affect migration of key hazardous components, methods of containment, or methods of remedial action clean-up.
- develop viable remedial action alternatives.
- permit the evaluation of the remedial action alternatives.
- recommend the most cost-effective technically feasible remedial option which has the ability to reduce impacts on human health, welfare and the environment to an acceptable level.
- prepare a conceptual design of the recommended remedial action alternative.

PROJECT ORGANIZATION

Attachment 1 lists the primary contacts for the project. Project technical personnel and quality assurance personnel are indicated in the project organization chart (Attachment 2). Primary responsibility for project quality review rests in the NWR Resource Contaminants Assessment Coordinator. Independent quality assurance review is provided by the Columbia National Fisheries QA/QC representatives, the refuge manager, and the USEPA On-Scene Coordinator.

QUALITY ASSURANCE OBJECTIVES

The general quality assurance objective for analyzed measurement data is to ensure that environmental monitoring data of known and acceptable quality are provided.

For this project, the specific objectives for measurement data in terms of precision, accuracy and compatibility are the same as the objectives established for the Statement of Work for the U.S. EPA Contract Laboratory Program (CLP), viz.: "The purpose of the QA/QC program....is the definition of procedures for the evaluation and documentation of subsampling, analytical methodologies, and the reduction and reporting of data. The objective is to provide a uniform basis for subsampling, sample handling, instrument condition, methods control, performance evaluation, and analytical data generation and reporting." This QAPP for sampling, analysis and data handling is consistent with the requirements set forth by the U.S. Fish and Wildlife Service, as well as all State and Federal EPA requirements.

SAMPLING PROCEDURES

The objective of sampling procedures is to obtain samples that represent the environmental matrix being investigated. Trace levels of contaminants from external sources will be eliminated through the use of good sampling techniques and proper selection of sampling equipment.

A detailed description of sampling procedures is presented in the Site Sampling Plan (Appendix B). Source material used in developing the sampling plan included the following:

Technical Support Documents

- Samplers and Sampling Procedures for Hazardous Waste Streams (EPA-600/2-80-180)

- Test Methods for Evaluating Solid Wastes (EPA SW 846-1980)
- User's Guide to the EPA Contract Laboratory Program
- EPA Technical Monographs
  - 15--Purposes and Objectives of Sampling
  - 16--Water Sampling Methods
  - 17--Soil and Sediment Sampling Methods
  - 18--Sampling of Biological Specimens
  - 19--Methods of Collecting Concentrated (Hazardous) Samples
  - 20--Container Opening Techniques
  - 22--Sample Handling, Packaging, and Shipping Procedures

The Site Sampling Plan (Appendix B) includes the following protocols and documentation.

- Number of locations to be sampled
- Sampling procedures to be used at the site
- Tests to be completed at each sampling location
- Sampling equipment required at the site
- Sample containers required at the site
- Preservation methods to be used at the site for various types of samples
- Reagents, etc., required at the site for sample preservation
- Shipping containers required at the site
- Chain-of-custody procedures to be used at the site
- Shipping methods and destinations, marking instructions, special labels, etc.

SAMPLE CUSTODY

Sample custody procedures for this project will be in strict conformance with the procedures detailed in NEIC Policies and Procedures (EPA-33019-78-001-R.) These procedures were established to comply with EPA requirements for sample control. They are documented in Attachment 4 to this QAPP.

All samples collected for analysis will be taken by chemists, physical science technicians, or other qualified personnel designated by O'Brien & Gere with specific instructions from the Project Manager. The FWS will take duplicate samples at a ratio of 1:10 for QA/QC purposes. All samples for residue analysis will be placed in the custody of the analytical chemist responsible for the analysis. The sample information will be recorded on the same report sheets if analyzed immediately. Stored samples (including archive portions) will be catalogued and stored appropriately for future analysis. The record of samples cataloged and stored may be audited by the QA Officer. Subsequent to approval of the conceptual design (Task 15), these archived samples will be returned to CONWR for disposal consistent with the remedial action plan.

EQUIPMENT CALIBRATION, REFERENCES AND FREQUENCY

All field equipment used during this project will be calibrated and operated in accordance with manufacturer's instructions. Any field equipment used during this project that is not covered by the investigator's standard operating procedures will have a specific calibration

and operation instruction sheet prepared for it. The specific instruction sheet(s) are on file with the analytical laboratory. See also Attachment 4 to this QAPP.

A. General

Standards may be generally grouped into two classifications: primary and secondary. Primary standards include USP and NF drugs, NBS and ASTM materials, and certain designated EPA reference materials. All other standards are to be considered secondary.

B. Testing

1. Primary: No testing is necessary. Do not use if there is any physical indication of contamination or decomposition (i.e. partially discolored, etc.).
2. Secondary: Examine when first received either by comparison to an existing primary, or comparing known physical properties to literature values. The less stable standards will be rechecked at appropriate intervals, usually six months to one year.

C. Storage

1. All standards will be stored in appropriate locked areas.
2. All special storage requirements (i.e. refrigeration, storage under nitrogen, etc.) will be met.

D. Records

1. A records book will be maintained for each grouping of standards (i.e. pesticides, metals, etc.)
2. The record kept for each standard will include:
   a. Name and date received
   b. Source
   c. Code or lot number
   d. Purity
   e. Testing data including all raw work and calculations
   f. Special storage requirements
   g. Storage location
3. These records will be checked periodically as part of the Laboratory Controls Review.

EQUIPMENT

The following protocols are further documented in the Laboratory QA/QC manual (Attachment 4 to this QAPP).

A. General

1. Each major piece of analytical laboratory instrumentation used on this project is documented and on file with the analytical laboratory.
2. A form is prepared for each new purchase and old forms will be discarded when the instrument is replaced.

B. Testing

1. Each form details both preventative maintenance activities and the required QA testing and monitoring.

2. In the event the instrument does not perform within the limits specified on the monitoring form, the Laboratory Manager will be notified and a decision made as to what action to take.
3. If repair is deemed necessary, an "out of order" sign will be placed in the instrument until repairs are effected.

C. Records

1. A bound notebook is kept with each instrument to record all activities related to maintained, QA monitoring and repairs.
2. These records will be checked during periodic equipment review.

DATA ANALYSIS

All raw data collected from project sampling tasks and used in project reports will be appropriately identified and will be included in a separate appendix within the RI report. Data will be reported in units in accordance with industry standards. Where test data have been reduced, the method of reduction will be described in the report.

Data will consist of raw output such as chromatograms, computer assisted integrations of the chromatograms, extraction, routing and quantitation sheets as well as quality control summaries. The raw data will be processed and compiled into a finished data summary. The finished data summary will then be submitted to the Project Manager who will arrange for transfer of information to FWS. All raw data will be filed and archived by O'Brien & Gere and made available for review on request.

QUALITY CONTROL PROCEDURES

Quality control of data will involve the collection of field sample duplicates and blanks in accordance with the applicable EPA Technical Monograph 15 through 22. The standard quality control procedures established for the CLP will be employed to provide consistent, accurate, and dependable test results.

Attachment 4 to this QAPP documents the QA/QC considerations included in this program. The major elements of the QA/QC program are: instrumental tuning and calibration criteria, defined analytical protocols, reagent blanks, surrogate spikes, matrix spikes and duplicate analyses. A reagent blank is included in each batch of up to twenty samples analyzed. Surrogate spike standards are incorporated into all samples and blanks prior to sample processing while one set of matrix spikes and matrix spike duplicates will be included per batch of up to twenty samples. A field blank consisting of diatomaceous earth for soils or distilled water for groundwater will also be included as quality control samples.

AUDIT PROCEDURES

The O'Brien & Gere Project Manager, the Columbia National Fisheries QC/QA Representative and the Refuge Manager will monitor and audit the performance of the QA procedures listed in this plan. They will conduct field and office audits.

O'Brien & Gere has designated a QA officer as outlined on Attachment 2 to this QAPP. A performance audit, consisting of analysis of appropriate blanks, fortified samples and standard solutions will be performed quarterly for the duration of the project. O'Brien & Gere's

QA Officer will maintain a record of such audits and will inform the FWS of significant deviations from established control limits. These audits will test not only the total system's response, but inherently all major measurement methods.

O'Brien & Gere's QA Officer will report to the Project Manager and the FWS the result of assessment of: the accuracy, precision and completeness of the data, results of the performance and system audits, and any problems encountered in the analytical procedures. The QA Officer, in conjunction with the analyst, analyst's supervisor, and Project Manager will formulate recommendations to correct any deficiency in the analytical protocol or data. These corrective measures will be in accord with ongoing good laboratory practices and the overall Quality Assurance Program.

ANALYTICAL PROCEDURES

All samples collected during this project will be delivered to the laboratory for analysis in accordance with the standard analytical procedures established by the EPA for the Contract Laboratory Program and as documented in Attachment 4 to this QAPP.

The analytical protocols to be used for specific analyses included in this program are identified on Attachment 3 to this QAPP. Samples analyzed for chlorinated dioxins and dibenzofurans will be analyzed according to the procedure of Smith et al. (1984) or equivalent as presented in Attachment 5 to this QAPP. Analytical procedures for explosives in soils are presented in Attachment 6 to this QAPP.

The listing of specific analyses to be performed on each sample taken from various sites around the Refuge is included in Attachment 1

to Appendix B (SSP). For priority pollutant organics and PCDD/PCDF, several samples will be subjected to the screening only. The screening is the first step in analysis of organics by the CLP protocol which indicates whether organics are present within the high range or low range. This will permit selection of a limited number of samples to be analyzed by the full CLP protocol.

CHECK SAMPLES

The FWS shall provide samples of soil containing known amounts of polychlorinated biphenyls (PCBs), PCDFs and PCDDs or priority pollutants to O'Brien & Gere. These samples will serve as Quality Assurance Samples. Quality Control Samples shall constitute approximately 1 in 20 samples from Area 9. O'Brien & Gere will advise the FWS of the analytical results promptly and FWS shall advise if remedial action is required.

PREVENTIVE MAINTENANCE

Preventive maintenance procedures will be carried out on all field equipment in accordance with the procedures outlined by the manufacturer's equipment manuals. Any field equipment used during this project that is not covered by the standard operating procedures will have a specific maintenance instruction sheet prepared for it.

DATA ASSESSMENT PROCEDURES

Analytical data will be submitted to and assessed by the FWS in accordance with their standard procedures. Analytical data will be

assessed based on laboratory performance for meeting instrument tuning criteria, surrogate recovery, duplicate analysis and reagent and field blank integrity.

CORRECTIVE ACTION PROCEDURES

Corrective action procedures that might be implemented from audit results or upon detection of data unacceptability are developed on a case-by-case basis. Such actions may include altering procedures in the field, using a different batch of containers, or recommending an audit of laboratory procedures. Further guidance to corrective actions is outlined in Attachment 4 to this QAPP. The O'Brien & Gere Project Manager is responsible for initiating the corrective action. The Regional Resource Contaminants Assessment Coordinator is responsible for approving the corrective action.

QUALITY ASSURANCE REPORTS

For this project, no separate report is anticipated to describe the performance of the data measurement systems or the data quality. Discussions of quality assurance problems and corrective actions taken will be included in the project monthly progress reports. The final RI report and the final FS report will contain separate QA sections that summarize data quality information collected during the project.

ATTACHMENT 1

PRIMARY CONTACTS

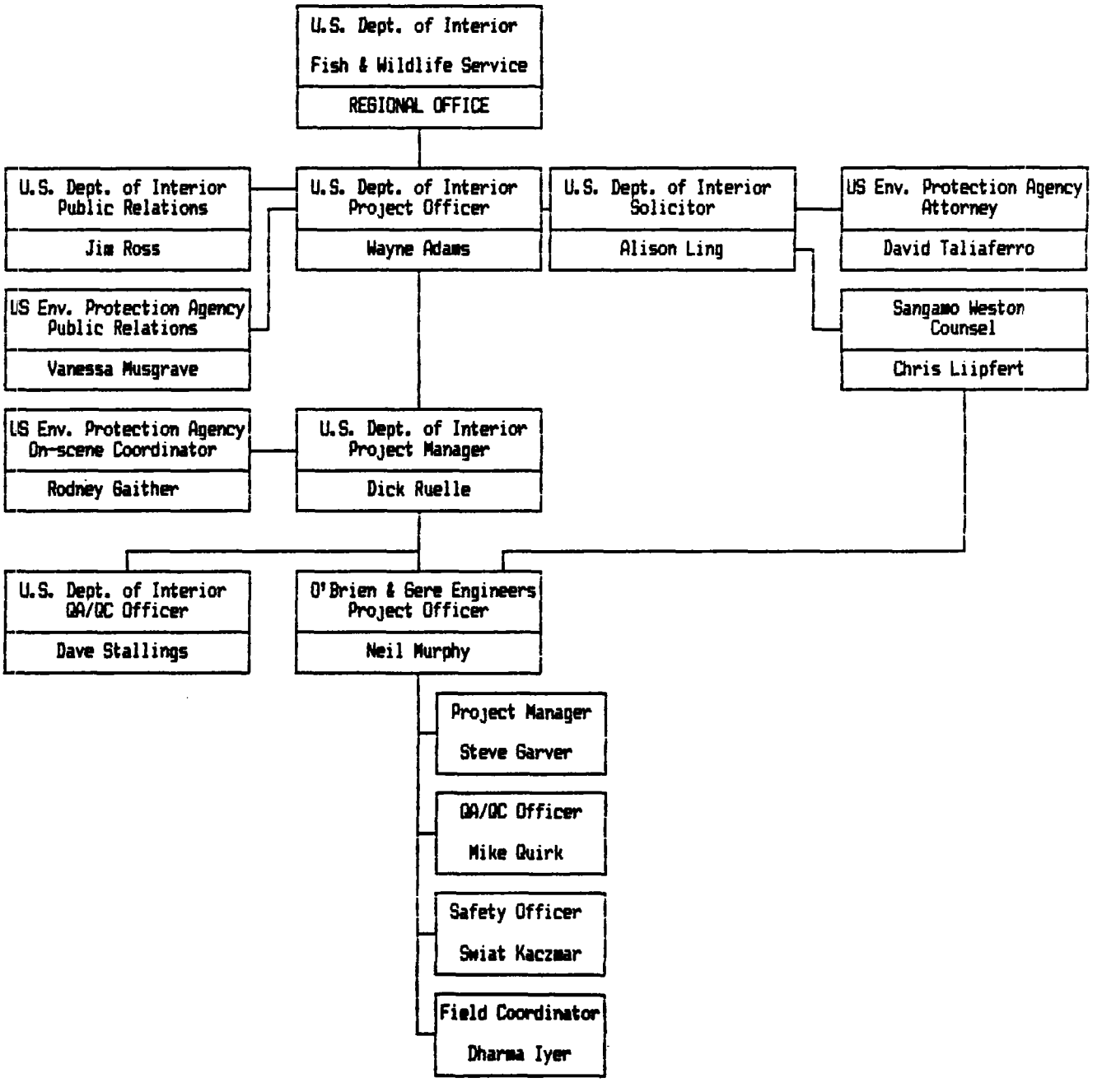
PRIMARY CONTACTS

Name and Responsibility	Organization and Address	Phone Number
Dr. James Elder Regional Resource Contaminants Assessment Coordinator	U.S. Fish and Wildlife Service Federal Building, Fort Snelling Twin Cities, MN 55111	612/725-3536
Mr. Wayne Adams Refuge Manager	U.S. Fish and Wildlife Service Crab Orchard National Wildlife Refuge P.O. Box J Carterville, IL 62918	618/997-3344
Dr. Dave Stallings Dr. Jim Petty Quality Control/ Quality Assurance	Columbia National Fisheries Research Laboratory U.S. Fish and Wildlife Service Route 1 Columbia, MO 65201	314/875-5399
Mr. Dick Ruelle Illinois Resource Contaminants Assessment Coordinator	U.S. Fish and Wildlife Service 1830 Second Avenue Rock Island, IL 61201	309/793-5800
Contracting and General Services	U.S. Fish and Wildlife Service Federal Building, Fort Snelling Twin Cities, MN 55111	612/725-3580
Mr. Rodney Gaither On-Scene Coordinator	U.S. Environmental Protection Agency 230 South Dearborn Street Chicago, IL 64604	312/886-4735
Mr. Bob Cowles Superfund Coordinator	Illinois Environmental Protection Agency 2200 Churchill Road Springfield, IL 62706	217/782-6760
Mr. Joe Stuart Illinois EPA Representative	Illinois Environmental Protection Agency 2209 West Main Marion, IL 62959	618/997-4371
Mr. Mike Carter Illinois Dept. of Conservation Representative	Regional Fish & Wildlife Manager Illinois Dept. of Conservation R.R. 4, Box 68 Benton, IL 62812	Office: 618/435-8138 Home: 618/883-5961

Ms. Vanessa Musgrave Community Relations	U.S. Environmental Protection Agency 230 South Dearborn Street Chicago, IL 64604	312/886-6128
Mr. Jim Ross Community Relations	U.S. Fish and Wildlife Service Federal Building, Fort Snelling Twin Cities, MN 55111	612/725-3519
Dr. Robert L. Flentge Illinois Dept. of Public Health Contact	Illinois Dept. of Public Health 525 West Jefferson Springfield, IL 62707	217/785-2439
Mr. Les Frankland Illinois Dept. of Conservation	Illinois Dept. of Conservation 424 Lincoln Tower Plaza Springfield, IL 62706	217/782-6424
Ms. Carol B. Luly Community Relations	Illinois Environmental Protection Agency 2009 Mall Street Collinsville, IL 62234	618/345-6220
Ms. Alison Ling Office of Soliciter U.S. Department of Interior	U.S. Department of the Interior Room 4354 18th & C Streets, N.W. Washington, D.C. 20240	202/343-5301
Mr. David M. Taliaferro Attorney, U.S. EPA	U.S. Environmental Protection Agency 230 South Dearborn Street Chicago, IL 64604	312/886-6826
Dr. Cornelius B. Murphy, Jr. O'Brien & Gere	O'Brien & Gere Engineers, Inc. P.O. Box 4873 1304 Buckley Road Syracuse, NY 13221	315/451-4700
Mr. John Hanson Beveridge & Diamond	Beveridge & Diamond, P.C. 1333 New Hampshire Ave., N.W. Washington, D.C. 20036	202/828-0285
Mr. Christian E. Liipfert Sangamo Weston, Inc.	Sangamo Weston, Inc. P.O. Box 48400 Atlanta, GA 30362	404/449-9006

PROJECT ORGANIZATION

Remedial Investigation/Feasibility Study
Crab Orchard National Wildlife Refuge



ATTACHMENT 3

ANALYTICAL PROTOCOLS

Parameters	Screening	Full Analysis
1. Purgeable Priority Pollutants	a) EMSL/LV : SEC. III D-54	a) EMSL/LV SEC. IV D64
2. Acid Extractable Priority Pollutants	a) EMSL/LV : SEC. III D-58	a) EMSL/LV SEC. IV D96
3. Base/Neutral Extractable Priority Pollutants	a) EMSL/LV : SEC. III D-58	a) EMSL/LV SEC. IV D96
4. Pesticide/PCB Priority Pollutants	b) METHOD 608	a) EMSL/LV SEC IV. D110
	Water	Soil
5. PCB's	b) Method 608	c) Method 8080
6. Metals		
- ICP scan	b) 40 CFR 136-App. C	c) Method 1010
- Priority Pollutant Metals by AA Spec.	d) EPA-Section 200	d) EPA - Section 200
7. Cyanide	b) 40 CFR 136-335.2	c) Method 9010
8. Indicators		
- pH	b) 40 CFR 136-150.1	c) Method 9041
- Specific Conductance	b) 40 CFR 136-150.1	c) Method 9050
- Total Organic Carbon	b) 40 CFR 136-415.1	c) Method 9060
- Total Organic Halogens	b) 40 CFR 136-410.1	c) Method 9010
9. Explosives Residues by HPLC	--	USATHAMA 84
10. Nitrogen Series: TKN, NH3N, NO3N	b) 40 CFR 136	--
11. PCDD/PCDF (Screening and Full Analysis)	Attachment 5	Attachment 5
12. Cation Exchange Capacity		
13. Total Phosphorus	d) EPA-365.1	d) EPA-365.1
14. Primary and Secondary Drinking Water Standards	40 CFR 141, 143	--

References

a) USEPA CONTRACT LABORATORY PROGRAM, EMSL/LV, Jan. 1985; "Organic Analysis Multi-Media, Multi-concentration."
b) Federal Register, 40 CFR Part 136, Oct. 26, 1984.
c) TEST METHODS FOR EVALUATING SOLID WASTE, USEPA, SW-846 (July 1982).
d) Methods for Chemical Analysis of Water and Wastes: EPA March 1979.

ATTACHMENT 4

LABORATORY QUALITY ASSURANCE/ QUALITY CONTROL PROGRAM

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I. O'BRIEN AND GERE LABORATORY

Introduction

For several years the O'Brien and Gere laboratory has been involved in the physico-chemical and microbiological analyses of environmental contaminants for federal, state, municipal and industrial clients. The laboratory has analyzed over 10,000 samples for over 100,000 parameters on an annual basis. The organic and inorganic pollutants occur in several matrices, i.e., potable water, industrial and domestic wastewater, hazardous waste, sludges, sediment, biological tissue, solid, air, etc. The ability to accurately characterize the chemical pollutants in these matrices is paramount.

In this document concepts are presented to outline the laboratory program purpose, policies, organization and operations established to support physico-chemical analyses conducted under USEPA compliance. Implementation of this program will better insure the validity of the data acquisition, and, therefore, will provide a more reliable foundation on which to base decisions. The principles and procedures used are the result of considerations of the general operations and trends in the field of analytical chemistry, analytical instrumentation, statistical quality control techniques, and previous experiences in the laboratory programs conducted under USEPA, local and state government compliance.

Laboratory Policy

The management of O'Brien & Gere's Laboratory is firmly committed to the Quality Assurance/Quality Control (QA/QC) program depicted in this manual. The program has been implemented and is maintained to assure any data reported by the laboratory are of known and documented quality commensurate with their intended use. The technical personnel who contribute to all or any portion of the laboratory analyses follow the procedures delineated in this manual.

The QA/QC manual is an integral part of a generalized representation of our Good Laboratory Practice program. It is primarily intended to set control guidelines and direction for all the physico-chemical and microbiological measurements performed by the laboratory. The contents of this manual will be re-evaluated yearly by the QA/QC group leader, and if necessary, revisions will be made, and/or the QA/QC program expanded.

A supplementary laboratory manual dealing with specific technical areas has been written and is available to all laboratory personnel. The laboratory manual is reviewed and approved by the QA/QC, Trace Organics and Wet Chemistry group leaders and management prior to distribution to the laboratory staff.

Quality Control Program Objectives

The primary objective of the O'Brien & Gere Laboratory QA/QC program is to assure the precision and accuracy of all data generated by the laboratory personnel. That is, the data is of known and documented quality.

The QA/QC guidelines are implemented in support of the laboratory surveillance programs and analyses efforts. They reflect the best cost effective effort, and are used to assess, ensure and document that all data collected, stored, reported or used by the laboratory are scientifically valid, defensible and of known precision and accuracy.

The major effort of the QA/QC program will be to develop a workable day-to-day "QA/QC model", and thus provide the detailed control charts and control limits to measure the laboratory daily performance. The QA/QC activities shall be carried out in accordance with EPA, state and local government mandates. The implementation, coordination and supervision of these procedures will provide the customer with the quality assurance (QA) activities associated with good laboratory practices.

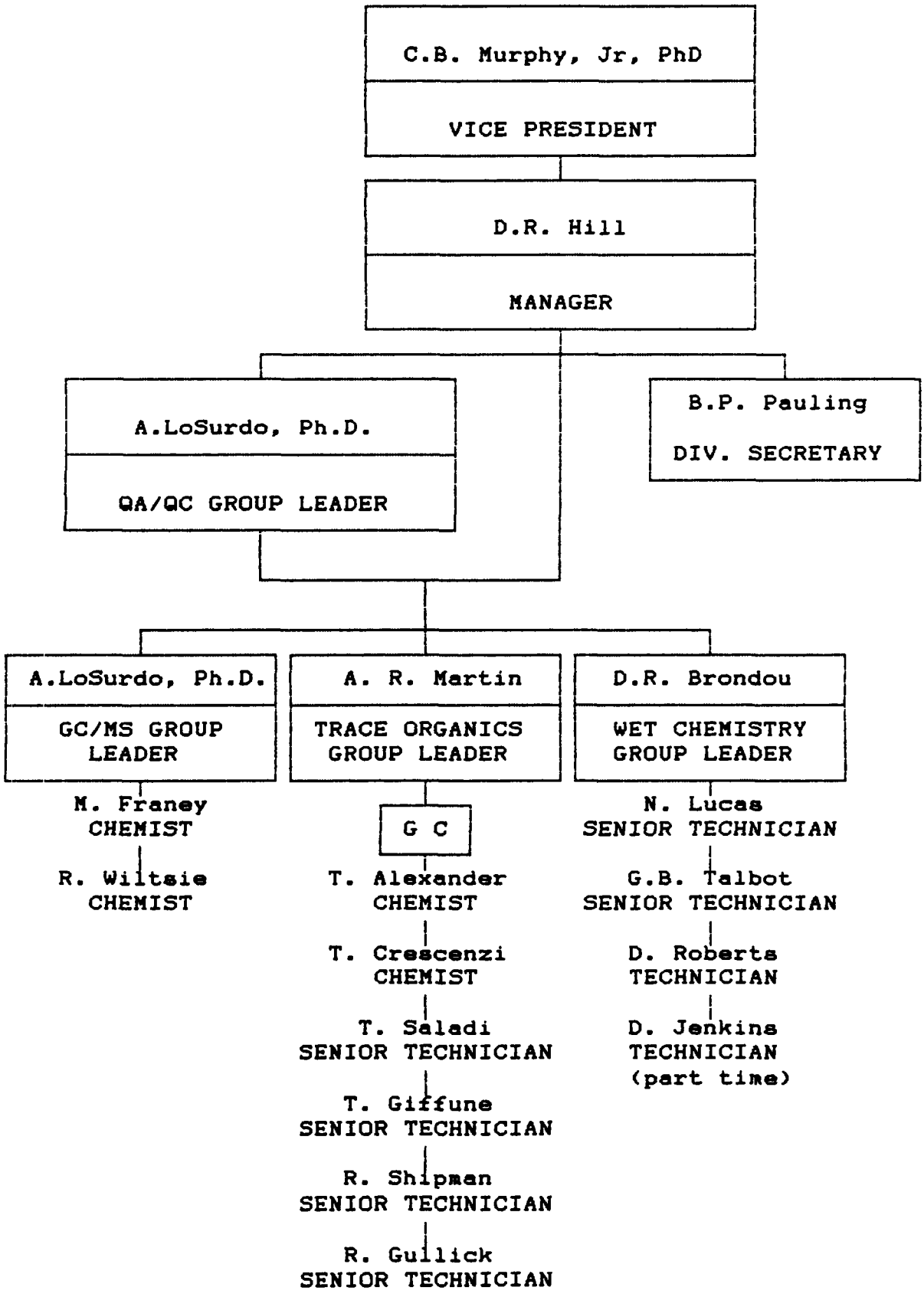
Personnel and Organization

Any organization consists of a number of people whose skills and delegated responsibilities assure the quality of the ultimate product, i.e. analytical services. QA/QC procedures commence when the sample is first collected, and continues until the final product is in the client's hand. An organizational chart of the laboratory technical staff is included in Figure 1 to serve as a frame of reference for all QA/QC procedures.

The Laboratory Manager is responsible for the overall administration of the analytical operations at O'Brien & Gere. The section group leaders handle the day to day scheduling and operation, and report to the manager. Together with the group leaders they review

FIGURE 1

LABORATORY ORGANIZATION CHART



and approve all policies concerning their specific areas of responsibility.

The QA/QC group leader is responsible for the implementation, monitoring and supervision of the QA/QC program. He assures that the program is conducted in strict adherence to procedures and requirements outlined in this manual. He reports to the Laboratory Manager, and interacts daily with other group leaders and laboratory staff. His duties include:

1. Develops and implements new QA/QC programs, including statistical techniques and procedures.
2. Conducts regular inspections and audits of analytical procedures.
3. Daily monitors accuracy and precision and implements correction measures if "out of control".
4. Maintains copies of all procedures routinely used in the laboratory measurements.
5. Informs management of the status of the QA/QC program by annual status reports.
6. Coordinates and conducts investigations of any customer complaints regarding quality.
7. Reschedule any analysis based on poor accuracy or precision data.

The section group leaders are responsible for the day to day operation and technical questions concerning analytical protocol and together with the QA/QC group leader:

1. Maintain and increase the technical skills of the laboratory technical personnel to achieve optimum quality results.

2. Approve analytical methods, sampling procedures, special QA/QC procedures, and any subsequent revisions in analytical procedures used in their respective areas.
3. Approve completed work.

Technical Training

All personnel involved in any function affecting data quality (sample collection, analysis, data reduction, and quality assurance) have sufficient technical training (in their appointed positions) to contribute to the reporting of complete and high quality data. The training is achieved through: a) On-the-job training, b) Short-term courses (one week or less), and c) Long-term courses (one semester or longer).

Short and long term courses are available through universities, colleges, and technical schools in statistics, analytical chemistry, and other disciplines. In addition, short-term courses are provided by commercial training organizations, manufacturers of equipment and others.

The trainee and/or analyst performance is evaluated by providing unknown samples for analysis. An unknown, as defined here, is a sample whose concentration is known to the QA/QC group leader or other group leaders but is unknown to the trainee or analyst. Proficiency is judged in terms of accuracy.

II. GENERAL FACILITIES AND EQUIPMENT

The laboratory is located in the corporate headquarters of O'Brien & Gere in Syracuse, The laboratory maintains a staff of sixteen chemists, biologists and technicians. As many as ten temporary and part-time personnel have been used to meet peak demands. The staff maintains a constant awareness of state-of-the art techniques in environmental analysis through its review of literature. The laboratory has 3700 square feet to utilize for the preparation and analysis of samples and 1200 square feet for receiving and storage of reagents.

The laboratory's involvement in a variety of programs has provided the necessary experience in microbiological, inorganic contaminants and trace organic identification and quantification. Particular expertise has been developed in the area of hazardous waste identification and trace organics analysis including priority pollutants and PCB's. A brief description of available instrumentation, computer services, sample storage and receiving follows.

Laboratory Instrumentation

The following analytical instrumentation is located in the Syracuse office and has been used on a number of major analytical programs:

(a) Hewlett Packard 5993B Gas Chromatograph/Mass Spectrometer Data System - for the low level identification of organic priority pollutants and other compounds. The unit is equipped with a dual disc, 32K computer and 9-track magnetic tape.

(b) Hewlett Packard 5880A - Gas Chromatograph equipped with dual electron capture detectors. The fully automated system has capabilities for both packed and capillary column work. The system can

operate unattended around the clock to provide rapid turnaround of results.

(c) Tracor Model MT220 gas chromatograph equipped with electron capture and dual flame ionization. The unit is interfaced to a Hewlett Packard Model 3380 S integrator.

(d) Two Tracor Model 550 gas chromatographs, both equipped with Hall electrolytic conductivity detectors, linearized electron capture detectors, and photoionization detectors interfaced to Hewlett Packard Model 3390 integrators.

(e) Due to the highly specialized procedures for cleaning glassware used in the low level analysis of halogenated organics and other substances, a sonic cleaner is utilized. Additionally, a complete glassware supply including Soxhlet extractors, separatory funnels, flasks and chromatographic columns is maintained.

(f) Two Technicon AutoAnalyzers, single and dual channel, for the automated determination of nutrients and other inorganic parameters.

(g) Perkin-Elmer Model 290B Atomic Absorption Spectrophotometer for the determination of metals by flame techniques.

(h) Varian Model 575 Atomic Absorption Spectrophotometer for the low-level detection of metals by conventional flame and graphite furnace (flameless) techniques.

(i) Beckman Model 915 Total Organic Carbon Analyzer, for the determination of organic, inorganic or total carbon.

(j) Dohrman Model DX-20 Total Organic Halide Analyzer, and Model MCTS 20/30 Elemental Analyzer for the determination of chlorine and sulfur in environmental samples.

(k) Bausch & Lomb Model 340 colorimeter, used for those colorimetric procedures not performed on the AutoAnalyzers.

(l) DuPont Model 760 Luminescence Biometer for the determination of adenosine triphosphate (ATP).

(m) Orion Model 4 Specific Ion Meter.

(n) Mettler Model HE10 Electronic Semi-Micro Balance.

(o) Hiack Particle Counter for the determination of particle sizes in water ranging from 0.5m to 300m.

(p) A walk-in refrigerator for storage of samples prior to analysis.

The laboratory also maintains a wide range of the usual supporting equipment such as pH meters, analytical balances, ovens and incubators, refrigerators and hood space.

Computer Services

The hardware which serves as the foundation of the firm's computer facilities has been responsible for the ability of the O'Brien & Gere laboratory to store and retrieve all data for individual clients.

The quantity of data has led to the development and utilization of a computer-based data management system. Samples are logged in, analyses are scheduled and output is received, all via time-shared or batch computer programs. One of the benefits of this system is that turnaround time has been reduced to a practical minimum. Data can be reported in a variety of formats. The standard computer output includes sample identification and various test results. A variety of statistical and modeling programs are available for the evaluation and interpretation of data.

III. GENERAL CONSIDERATIONS

Maintenance

A preventative maintenance schedule on all instruments, balances, and equipment requiring maintenance is followed. All maintenance, whether performed by the laboratory or other professional sources, is documented in appropriate log books. Entries are made each time maintenance is performed and include the reason for maintenance, what was performed, by whom, and the dates and initials of the analyst in charge during the maintenance.

Calibration

Thermometers needed for critical temperature determination and control are calibrated against an NBS thermometer on site once a year. Analytical balances are professionally calibrated and cleaned once a year and checked with Class S weights daily by analysts who routinely use the balances. Calibration data are entered into a specific calibration notebook, which is kept with the equipment being calibrated. When the balances are professionally calibrated, a document stating the specific balance (model and serial number), its location, and the data calibrated is provided by the company or individual providing such service.

Reagent Quality

The quality of reagents and instrument readings are maintained by the following procedures:

(a) Reagents for quantitative purposes are ACS analytical quality grade or better.

(b) Each sample is collected in a new container to minimize contamination. This rule does not apply to bacteriological samples for which sterilized glass bottles are used, or trace organic samples for which solvent rinsed glass bottles are used.

(c) Distilled deionized water with a conductivity not more than 1.5 micromho/cm is used in the preparation of all reagents and for final rinses. The conductivity is measured daily and recorded in the quality control log. The pH is also checked daily and the values recorded.

(d) All volumetric glassware is National Bureau of Standards Class A grade or better.

(e) All glassware is cleaned and rinsed with distilled water and visually inspected before use. Any volumetric glassware found to be etched or cracked is discarded.

(f) The operating temperatures of all ovens, incubators, water baths and refrigerators are recorded daily in the quality control log.

(g) All reagents are discarded after a set interval which has been established and recorded in the Laboratory Handbook.

(h) The date a prepared reagent is made is entered into the Reagent Log and initialed by the preparer. Therefore, the results which have been affected by a contaminated or otherwise improper reagent can be easily determined. These results are either recalculated or discarded and the analysis may be repeated if possible. Reagent containers are also dated when new solutions are prepared and are initialed. These procedures are followed for all (even daily) preparations.

(i) The pH meter is checked with three buffers (4.0, 7.0 and 10.0) and the results are recorded in the quality control log.

Safety

A safety manual is issued to all laboratory personnel and describes safety policies, procedures and guidelines. Although laboratory workers are trained to be cautious in handling toxic or dangerous materials, they have confidence in the safety features built into their working area, thus enhancing the reliability of their performance.

Audits and Inspections

The Quality Assurance program is audited weekly for overall adherence to the guidelines and procedures outlined in this manual. The QA/QC group leader is responsible for scheduling and ensuring that each audit occurs.

Monthly meetings are scheduled between the QA/QC group leader and manager of Analytical Services to thoroughly discuss the program. Any corrective action required is monitored and ensured by the QA/QC group leader.

IV SAMPLE COLLECTION AND TRACKING

Valid representative samples of environmental matrices are collected through well defined sampling protocols. The sampling may be performed by the laboratory sampling team, or the customer who then assumes responsibility for properly obtaining, handling, preserving and shipping the sample.

Sample Collection and Handling

A well defined sampling protocol must ensure that:

a. sampling team members are competent and qualified

b. proper sampling methods are used

c. equipment is accurately calibrated

d. all samples are properly handled to prevent contamination

e. samples analyzed are actually the samples collected under reported conditions.

For these reasons, samples are kept in secure places from time of collection until they are analyzed. It is the joint responsibility of the group leader and sampling team leader to ensure that approved methods are used, and it is the responsibility of each sampling technician to assure that the equipment is accurately calibrated.

Chain of Custody

The laboratory sampling protocol generally follows a chain of custody procedure. The procedure creates an accurate, written, legally defensible document that can be used to trace possession of sample from its collection through analysis and final disposal.

The basic elements in the chain-of-custody phase of our QA/QC program are:

1. Sample collection and handling
2. Sample analysis
3. Preparation and filing of test report

These measures are documented by the chain of custody form (Figure 2) signed by all handlers of the sample(s). As defined here, a sample is "in custody" if it is:

a. in actual physical possession, or
b. in view after being in physical possession, or
c. in a locked repository, or
d. in a secure, restricted area.

Analysis, Preparation and Filing of Test Report

A critical concern of QA/QC program is the maintenance of sample and data base integrity and the timely preparation of data reports. The data management program allows for the identification of samples and the maintenance of the discrete character of the data generated by each respective sample. This system is a unique advantage over manual methods and has permitted the laboratory to successfully tabulate data involving high numbers of samples and multiple analyses. The system may be divided into the following phases:

1. sample identification -- as each sample enters the laboratory, it is assigned a unique access number found on a sample identification ticket. This identifier permits the discrete organization of all information and data relating to that sample, whether for analytical

FIGURE 2

CHAIN OF CUSTODY RECORD

SURVEY				SAMPLERS: *(Signature)*					
STATION NUMBER	STATION LOCATION	DATE	TIME	SAMPLE TYPE			SEQ. NO.	NO. OF CONTAINERS	ANALYSIS REQUIRED
				Water		Air			
				Comp.	Grab.				

Relinquished by: *(Signature)*	Received by: *(Signature)*	Date/Time
Relinquished by: *(Signature)*	Received by: *(Signature)*	Date/Time
Relinquished by: *(Signature)*	Received by: *(Signature)*	Date/Time
Relinquished by: *(Signature)*	Received by Mobile Laboratory for field analysis: *(Signature)*	Date/Time
Dispatched by: *(Signature)* Date/Time	Received for Laboratory by:	Date/Time
Method of Shipment:		

identification purposes, reference in paper-copy records and correspondence, or computer storage and recall.

2. data organization -- in a preliminary planning phase of any analytical investigation involving the laboratory, a computer codification format can be established which can serve as the basis for storage and retrieval of data. This format is characterized by the categorization of samples, with any type of identification permissible for the classification. The categories may be based on any similarities (or dissimilarities) in the total volume of samples.

The storage and retrieval of quality control sample data is also managed with the laboratory's computer-based data management system. Samples are tagged and data is input, stored and retrieved as with any routine project samples. This has been made possible by the use of a unique quality control project number by which such data may be identified.

V. METHODS AND PROCEDURES

The laboratory analyzes a variety of matrices for a number of different environmental constituents of concern. Therefore, several documents are referenced which include the procedures employed. The following list itemizes the most widely used documents:

1. Standard Methods for the Examination of Water and Wastewater.
2. Methods for Chemical Analysis of Water and Wastewater.
3. ASTM Annual Book of Standards.
4. Code of Federal Regulations.
5. NIOSH Manual of Analytical Methods.
6. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.

When analyzing samples by the above standardized methods, the accuracy or precision of the data generated by the laboratory is determined through analysis of replicates, spiked samples, synthetic reference standard samples, and/or field or laboratory blanks along with each set of samples. Any interferences are identified and documented.

In general, the methods accuracy is determining by spiking the sample matrix with the analyte at a minimum of three concentration levels. The range of the spiking levels is selected to bracket the concentration of interest. Percent recoveries of the spikes are calculated and are compared with synthetic standards. The methods precision is determined by analyzing a minimum of three replicates at each spiking level. The precision is evaluated by calculating the standard derivation.

The data generated is, whenever possible, input into the laboratory base data management system. Analyst's work sheets are filed for one year as a temporary record. When approved and signed, data reports and pertinent information are reported to the customer.

VI. INTRALABORATORY QA/QC PROGRAM

A quality control program is a systematic attempt to assure the precision and accuracy of analyses by detecting and preventing recurrency of errors, or measuring the degree of error inherent in the proven methods used. By identifying the sources of errors confidence in the precision and accuracy of analytical results can be established and improvements in the analytical methods made. To ensure the precision and accuracy of a result our quality control program requires the measurement and analysis of spiked samples, duplicate samples, synthetic standards and blanks.

Duplicate samples are used to provide assurance that the procedure is under control and to determine the statistical limit of uncertainty (i.e., precisions). Synthetic standards and spiked samples are used to determine the quantification of the laboratory accuracy.

In general, our quality control program incorporates the concepts of: a) calibration to attain accuracy, b) replication to establish precision limits, and c) correlation of quantitatively related tests (synthetic standards and spikes) to confirm accuracy.

The overall effectiveness of the program is dependent upon the evaluation of: a) equipment and instruments, b) current state of the art, c) precision of the analytical method itself, d) expected ranges of analytical results, e) control charts to determine trends as well as gross errors, f) data sheets and laboratory procedures adopted for control of sample integrity, g) quality control results on a daily as well as on varying time frames.

Definitions of Basic Terms

Before we discuss the standard operating practice for the QA/QC program some definitions are in order. These are:

1. Reagent Blank - The reagent (or method) blank is an aliquot of pure, organic free water (or organic reagents) used in the analysis of samples. It is generated by passing the clean matrices through the entire analytical procedure (including all glassware and other materials that come into contact with the sample). These blanks are analyzed along with the samples to verify that: a) qualitatively, no false positives occur, and b) quantitatively, concentrations are accurate and do not reflect contamination.

2. Field Blanks - These are water blanks sent from the laboratory to the sampling site and are returned to be analyzed in the same manner as the samples. If the samples are to be analyzed for purgeable organics, the analysis of field blanks provide a check on possible contamination of the samples by permeation of volatiles through the septum seal. If positive interferences occur the analytical results are rejected unless sufficient data can be obtained from these blanks to allow correction of results.

3. Duplicates - Duplicates are the result of splitting a field sample into equal amounts and are treated throughout as two unique samples. The results of duplicate (or replicate) analyses provide information on the overall precision of the analytical methodology. Quantitative results are obtained by calculating the relative percent difference (RPD) for each analyte in the sample matrix.

4. Spike - Spikes are the result of the addition of a known amount of analyte to a sample or a blank. The analytical results yield

a quantitative measure of accuracy (spiked blanks) or percent recovery (spiked samples). The measured accuracy reflects the best result which can be expected, whereas the percent recovery reflects matrix effects upon the analytical method accuracy.

Because several different environmental matrices are analyzed (e.g., potable water, effluent and influent waters, process wastes, sludges, etc.), two spiking levels are necessary when analyzing different samples. Relatively clean samples are spiked at detection limit and 10 times the detection limit for each component. Highly polluted samples are spiked at 100 times the detection limit for each component. Ideally, the spike should be 50 - 100% of the original concentration of each analyte in the sample matrix. If the added spike is less than 10% of the sample result, the data are questionable and statistically unacceptable.

5. Surrogate Spike - These are the result of the addition of known amounts of standards to every sample prior to the analysis. The standards are chemically similar to the compounds in the fraction being analyzed. In addition, some standards added have compounds which are not likely to be found in environmental samples. The analyses of surrogate spikes provide quality control on every sample by constantly monitoring unusual matrix effects, gross sample processing errors, etc. These spikes are not used as internal standards for quantitation.

6. Reference Standard (reference audits) - These are the analysis of independently prepared standard solutions or synthetic standards. Two types of standards are used, i.e., a) internal reference standard solutions (synthetic standards prepared in-house), and b) external

reference standard solutions obtained from outside sources (i.e., primarily EPA).

The external audits samples are used for monitoring the complete analytical method. These samples are introduced at the onset of the procedure (typically extractions) and carried through the entire analysis.

The internal standard audits are used to verify the "accuracy" of quantitative instrument calibration. All standard solutions are prepared by the QA/QC group leader and are submitted blind for analyses. The analyst analyzes the solutions as discrete samples and a percent recovery or percent error is calculated. Errors greater than 5% are carefully investigated and differences resolved through proper action.

Guidelines for Evaluating the QA/QC Program

This section defines the QA/QC program for the analysis of environmental pollutants, i.e., the analysis of trace organics by gas chromatographic (GC) and GC/MS techniques, and analysis of inorganic pollutants by wet techniques and atomic absorption (AA), etc. The QC program for the analysis of trace organics by GC and GC/MS is different due to the unique nature of the analytical problems addressed by the GC/MS methodology. Therefore, the QC requirements for these two techniques will be addressed separately. A description of the QC program follows.

1. Gas Chromatography

In general, when GC methodologies are used the specific analyte or class of analyte is known. As a result a more specific, less generalized QC program can be defined. For example, accuracy data can be

collected prior to analysis of actual samples, and often previous QC data for a particular analyses is available.

The QC program outlined below depicts the procedures used to determine the quality of the data generated in the trace organics analyses. The steps monitored include extractions, concentration, qualitative and quantitative analyses and confirmation.

a) Method Verification

The methods are validated before they are used in routine analysis of samples. Method validation includes analysis of reagent blanks, blanks spiked with compound(s) of interest, analytical standards and standard mixtures. The results from these analysis approximate the best data to be expected from the method.

The extraction and concentration steps are validated by spiking a minimum of 2 blank samples with the same matrix as the sample of interest. The concentration of the analyte used for the spiking is 10 times the detection limit. The accuracy (or percent recovery) of the method is calculated by:

$$\text{ACCURACY} = \frac{\text{(spiked sample result)}}{\text{spike added}} \times 100$$

and is recorded on transcription sheets and is assigned a unique QC number. The data is then logged and stored in the computer.

b) Instrument Calibration and Performance

To insure good analytical data the analytical instruments are calibrated prior to sample analysis by analyzing three standards of analyte which span the suspected concentration range of the analyte in the sample. The performance of the instruments are checked by analyzing a standard mixture. If the retention time or

area counts vary more than 10% from previous calibration the standard mix is reanalyzed. If the deviation is still more than 10%, a new standard mix is analyzed. If the new standard mix still yields greater than 10% deviation, instrument malfunction is suspected and proper action is taken to resolve the problem.

Routine Analysis

The quality of the analytical data generated during routine analyses is monitored by the following:

1) Contamination from reagents and glassware is identified by analyzing a reagent blank. One reagent blank is prepared for every 20 or fewer samples analyzed (or when a new lot of reagent is used in the analysis).

2) The analytical method accuracy is determined by spiking a known amount of analyte into a sample and blank. The percent recoveries are then calculated. The amount of analyte recovered from the blank indicates the best result which can be expected from the method. The amount of analyte recovered from a sample reflects matrix effects upon the accuracy of the method. Two spikes are prepared for every 20 or fewer samples analyzed.

3) The analytical method precision is determined by analyzing equal amounts of a split sample. Ideally, the analytical results will be identical; however, differences occur due to variations in the procedure. A quantitative measure of these differences is assessed by calculating the relative percent differences (RPD) for each analyte in the matrix and the results compared.

In general, one duplicate is analyzed for every 20 or fewer samples, and the performance of the analytical instrument verified. Whenever possible identification is confirmed by a second procedure.

GC and GC/MS Characterization of Trace Organics

The requirments for the characterization of trace organics analyses include: 1) the identification and quantitation of unknown pollutants, 2) the specific detection of selected groups of pollutants (i.e., Priority Pollutants by GC/MS), and 3) other analyses requiring GC/MS for identification, verification and/or quantitation. A summary of the required audits is given in Table I. The performance and calibration of the GC and GC/MS systems are monitored and maintained on a regular basis by the procedures and methods discussed below.

TABLE I. SUMMARY OF SAMPLE ANALYSIS AUDITS REQUIRED FOR THE CHARACTERIZATION AND QUANTITATION OF TRACE ORGANICS

AUDIT	AUDIT
Spike	Mass Spectrometer:
Reagent Blank	mass calibration
Duplicate Sample Analysis	response calibration
Standard Mix	standards
Reference Standard	Computer Match
Standards and Calibration Curve	Reference Spectra Comparison
GC Retention Times	Completeness and Accuracy
GC Peak measurement calculation	

1. Calibration of GC/MS System

At the beginning of each day the GC/MS system is calibrated and tuned by examining the mass spectrum of decafluorotriphenylphosphine (DFTPP) or 4-bromofluorobenzene (BFB). The details are discussed below.

a. Base/Neutrals (and Acids or Pesticide) Fractions

The analysis of 50 nanograms of DFTPP is carried out daily by direct injection into the GC inlet. The resulting mass spectrum is then examined. The requirement is that the mass spectrum of 50 nanograms DFTPP must meet the specification of the key ions and ion abundance criteria listed in Table II.

b. Volatile (Purgeable) Fraction

The analysis of 20 nanograms of BFB is carried out by direct injection into the GC/MS. The requirement is that the mass spectrum of 20 nanograms BFB must meet the prescribed specifications of the key ions and ion abundance criteria listed in Table II.

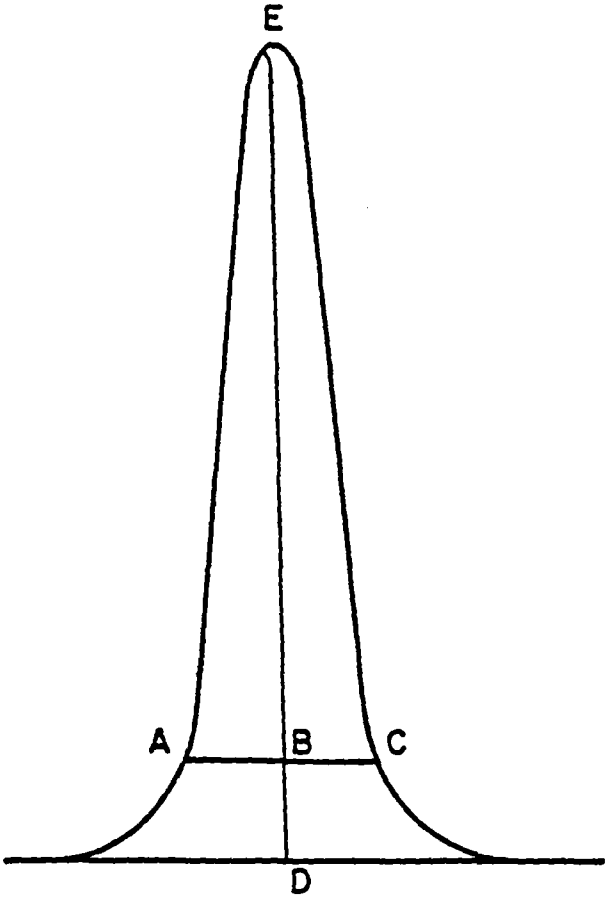
2. GC Column Performance Check

The GC columns performance are checked at the beginning of each day that samples are analyzed. For base/neutrals and acid fractions the columns performance are monitored by injecting 100 nanograms (ng) of benzidine and pentachlorophenol, respectively. For purgeables the column is checked by injecting 20 ng of BFB. Performance acceptance is based on calculations of tailing factors (see Table III).

TABLE II. KEY IONS AND ION ABUNDANCE CRITERIA FOR DFTPP AND BFB

DFTPP		BFB	
MASS	ION ABUNDANCE CRITERIA	MASS	ION ABUNDANCE CRITERIA
51	30-60% of mass 198	50	20-40% of mass 95
68	less than 2% of mass 69	75	50-70% of mass 95
70	less than 2% of mass 69	95	base peak, 100% relative abundance
127	40-60% of mass 198	96	5-9% of mass 95
197	less than 1% of mass 198	173	less than 1% of mass 95
198	base peak, 100% relative abundance	174	70-90% of mass 95
199	5-9% of mass 198	175	5-9% of mass 95
275	10-30% of mass 198	176	70-90% of mass 95
365	greater than 1% of mass 198	177	5-9% of mass 95
441	less than mass 443		
442	greater than 40% of mass 198		
443	17-23% of mass 442		

TABLE III EXAMPLE CALCULATION OF TAILING FACTOR



TAILING FACTOR = (BC/AB)

Example calculation:
Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm
Therefore: Tailing Factor = (12/11) = 1.1

Wet Chemistry and Bacteriology

The quality of the analytical data generated from inorganic and microbiological analyses of environmental contaminants are monitored as follows:

1. Wet Chemical Instrumental Methods

The atomic absorption (AA) spectrophotometer and AutoAnalyzer are calibrated using appropriate calibrating standards and blanks. The calibrations are checked by analyzing synthetic standards at five different concentration levels. The results are used to generate standard curves by least squares fit of the data via computer programs. The deviation of the standards from the least squares fit (standard curves) and the standard deviation of the fit are printed on the daily printout and the data stored accordingly in appropriate computer data bases. If deviation from accepted values occur analyses of sample and instrumental calibrations are repeated. Standard curves are generated regularly.

For colorimetric analyses that do not use the standard curve program, one or more standards are analyzed with each group of samples. The results are compared to generally accepted criteria, i.e., percent recovery (or percent error) and relative percent error.

Spectrophotometric instruments are checked by comparing the gain settings or percent transmittance for known (synthetic) standards to previous values. This monitoring method shows any decrease in sensitivity or other systematic effects in performance.

The conductivity meter is checked each time a group of samples is analyzed. The conductance of a standard solution is entered in the quality control log. In addition, the cell constant is checked annually by measuring the electrical conductivity of potassium chloride reference solution. The results are also entered in the quality control log book.

2. Bacteriology Techniques

Quality control extends to all aspects of the bacteriological laboratory. The date of preparation of media and the various solutions used in analysis are recorded in the quality control log together with any information which may be important to its preparation such as pH, lot or control number, manufacturer and concentration. In addition, random samples of prepared media are incubated under the same conditions as unknown samples to insure the maintenance of sterility during preparation and use.

The efficiency of autoclave sterility is monitored by the monthly use of Kilit ampules (BBL), a suspension of Bacillus stearothermophilus spores. The sterility of rinse water is checked periodically by the filtration and incubation of a reagent blank (sterile rinse water).

As part of the overall quality control program, the bacteriological quality of the distilled deionized water supply of the laboratory is monitored weekly. Samples for the standard plate count are taken from the water system prior to entry to the deionization cartridge (following

distillation), after deionization and from the storage tank. The results are recorded in the quality control log. Additionally, the Suitability Test as described in Standard Methods is performed on a yearly basis by an outside laboratory qualified to undertake this testing. Bacteriological samples are included in the duplicate analyses program described in the chemical section.

Humidity checks are performed monthly on Standard Plate Count petri dishes to determine percent moisture loss upon incubation.

VII. INTERLABORATORY QUALITY CONTROL

To indicate how well our laboratory is performing by comparison with other laboratories performing similar work, O'Brien & Gere Laboratory participates in a variety of proficiency and roundrobin tests. Successful performance in the proficiency analyses of samples results in the laboratory certification.

Certification

The U.S. Environmental Protection Agency certifies state laboratories to conduct their own intrastate program of certification for the proficiency of private laboratories in potable water analysis. The EPA only certifies private laboratories directly in those states which have not assumed primacy. In New York State, the certifying agency is the NYS Department of Health. The firm's laboratory was one of the first participants in the New York State program and has been certified for chemical, atomic absorption, bacteriological and gas chromatographic analysis of potable water since 1974. Laboratory certification has been extended to the State of Massachusetts and interm states in the State of New Jersey for potable water and wastewater testing requirements.

In addition, the laboratory participates in the round robin analyses of reference samples supplied by the EPA and in the analysis of commercially available reference samples.

VIII. DEFINITIONS OF STATISTICAL TERMS

The following statistical term definitions are used to identify statistical reports and evaluations:

a. Accuracy and Precision - Accuracy is a measure of the nearness of an analytical result, or a set of results, to the true value. It is usually expressed in terms of error, bias, or percent recovery (PR).

Normally the term "accuracy" is used synonymously with "percent recovery". It describes either the recovery of a synthetic standard of known value, or the recovery of known amount of analyte (spike) added to a sample of known value. The percent recovery (PR) or "accuracy" can be calculated by using:

1. standards: PR = (observed value/true value) x 100

2. spikes: $$PR = \frac{(\text{conc. spike + sample}) - \text{sample}}{\text{conc. spike}} \times 100$$

Precision refers to the agreement or reproducibility of a set of replicate results among themselves without assumption of any prior information as to the true result. It is usually expressed in terms of the deviation, variance, or range. Good precision often is an indication of good accuracy, however, one can obtain good precision with poor accuracy if systematic (determinate) errors are present in the method or instrument used. Systematic errors are either positive or negative in sign. Other analytical errors are indeterminate (random) errors. These are inherent in the analytical methods due to uncertainties in measurements.

b. Average - The average or arithmetic mean ($\bar{X}$) of a set of n values (Xi) is calculated by summing the individual values and dividing by n:

$$\bar{X} = \left[\sum_{i=1}^{n} X_i\right] / n$$

c. Range - The range (R_i) is the difference between the highest and lowest value in a group. For n sets of duplicate values (X_2, X_1) the range (R_i) of the duplicates and the average range ($\bar{R}$) of the n sets are calculated by:

$$R_i = \left| X_2 - X_1 \right|$$

and

$$\bar{R} = \left[\sum_{i=1}^{n} R_i\right] / n$$

d. Standard Deviation and Variation - The standard deviation (S) of a sample of n results is the most widely used measure to described the dispersion of a data set. It is calculated by using the equation

$$S = \sqrt{\frac{\sum_{i=1}^{n} (X_i - \bar{X})^2}{n-1}}$$

where $\bar{X}$ is the average of the n results and X_i is the value of result i. Normally, $\bar{X} \pm S$ will include 68% and $\bar{X} \pm 2S$ about 95% of the data in a normal distribution curve.

The variance is equal to S^2. The relative standard deviation (RSD) or coefficient of variation (CV) is the standard deviation divided by the mean and multiplied by 100, i.e.,

$$CV = 100S/\bar{X}$$

It is interesting to note that the precision is increased (value of S reduced) by increasing the number of duplicate analysis. The greater the number of replicate analysis, the greater the statistical confidence that the true mean lies within certain limits about the experimental mean.

e. Standard Calibration Curves - standard calibration curves are widely used in the analysis of inorganic pollutants. These curves are generated from the results of analyses of three or more standard solutions of known concentration and a blank. Typically, they are plots of the instrument response versus concentration. A plot is defined as linear, i.e., obeys the linear equation $Y=a + bX$, if the correlation coefficient (R) calculated from the linear regression analysis is 0.996 or greater.

The intercept (a), slope (b) and correlation coefficients (R_c) can be calculated from:

$$a = \frac{\Sigma X^2 \Sigma Y^2 - \Sigma X \Sigma Y}{n\Sigma X^2 - (\Sigma X)^2}$$

$$b = \frac{n\Sigma XY - \Sigma X \Sigma Y}{n\Sigma X^2 - (\Sigma X)^2}$$

$$R_c = \frac{\Sigma(X_i - \bar{X})^2 (Y_i - \bar{Y})^2}{\sqrt{\Sigma(X_i - \bar{X})^2 \Sigma(Y_i - \bar{Y})^2}}$$

We fit the analytical data to a linear regression analysis by using a computer program.

f. Absolute and Relative Errors - An absolute error is the difference between the experimental result and the true value. The relative error is the absolute error divided by the true value and multiplied by 100 to yield the percent relative error (PRE). When the true value is not known, the PRE is a measure of the difference (range) of a replicate analysis divided by the mean of the replicate value and multiplying by 100. That is, for duplicates

$$PRE = \frac{100\,|X_2 - X_1|}{(X_2 + X_1)/2} = \frac{100\,|X_2 - X_1|}{\bar{X}_j}$$

g. Skewness and Kurtosis - Skewness and kurtosis are the numbers used to understand the shape of a given curve. Our groups are data bases of spikes, duplicates, and knowns. The data points in these groups should fall within a normal curve. Aberrations from the normal curve are detected in values of skewness and kurtosis.

Skewness defines the symmetry of a curve. A symmetrical curve must have a skewness of zero. Positive or negative values denote lack of symmetry. Kurtosis defines the peakedness of a curve. A normal distribution curve will have a kurtotic value of 3. Peaked curves will have values greater than three, and broad flat curves will have values

less than 3. These values are monitored by the QA/QC group leader. When aberrant values are noted, the interpretation is usually related to very high or low QC values entering data bases or the persistence of patterns of consistently high or low QC values. It is the QA/QC coordinator's responsibility to research the causes of excessive values and patterns and, where possible, rectify the analytical conditions leading to them.

References

1) "Handbood for Anayltical Quality Control in Water and Wastewater Laboratories," March, 1979 (EPA-600/4-79-019)

2) "Manual of Analytical Quality Control for Pesticides and Related Compounds in Human and Environmental Samples," January, 1979 (EPA-600/1-79-008)

IX. STATISTICAL QUALITY CONTROL AND THE "DAILY QC MODEL"

Random (indeterminate) and systematic (determinate) errors are inherent in all analytical methods due to uncertainties in measurements. The measurement of physico-chemical and microbiological properties of pollutants in various environmental matrices involve uncertainties which cannot be entirely eliminated. The errors in these measurements, however, can be reduced to tolerable limits by examining and controlling the significant variables.

Additional errors, often unrecognized, are introduced by interfering chemical reactions and other undesirable physico-chemical effects. In many instances absolute values cannot be attained directly.

Although uncertainties cannot be reduced to zero, they can be minimized by using available statistical methods. Estimates of the accuracy (probable "true value") and precision (range of measurement error) can be made for the various analytical methodologies by analyzing blanks, duplicates, spikes and synthetic standards. After sufficient QC data are collected various statistical methods are used to evaluate the quality of data by calculating control and warning limits. A discussion of the statistical methods used follows.

Control Charts

Control charts provide the necessary tool for detecting quality variations in the various analytical methodologies used for the quantitation of environmental pollutants. They are a continuous graphic indication of the state of an analytical procedure with respect to quality, and assist in deciding when and how to take corrective action. The QC charts are generated for each pollutant from the statistical

evaluation of QC data. A minimum of 15 duplicates and spiked samples and/or synthetic standard analyses are required to generate a control chart.

The control limits (CL) on QC charts are paramount criteria for assessing the significance of variations in the analytical results. For instance, when the plotted QC indicators (i.e., percent recoveries, relative percent error, etc.) fall within these limits, the analytical methodologies used are under "control". If, however, a QC indicator value falls outside the CL's, there is an indication that some assignable cause is present which has thrown the system "out of control". Thus, control limits can be considered warning or action limits. They enable us to detect deviations in analytical procedures, and therefore, take corrective action before producing erroneous results (or results which exceed the absolute maximum tolerable limits).

Common practice set warning limits (WL) at ± 2 standard (S) deviations (95% confidence level of the normal distribution curve) and control limits (CL) at ±3S limits (99.7% confidence level of the normal distribution curve) on each side of the mean. The CL and WL are calculated from the QC data of duplicates analyses by using the equations and statistical factors listed in Table IV. These CL's and WL's include approximately the entire data set under "in control" conditions, and are equivalent to the commonly used ± 3S and ± 2S limits, respectively. The qualitative relationship between upper and lower control limits, upper and lower warning limits, and the mean is shown in Figure 3.

TABLE IV STATISTICAL FACTORS AND EQUATIONS FOR CALCULATING QC (X BAR AND R) CHART LINES[1]

Observations in Subgroup (n)	Factor			
	A_2	d_2	D_3	D_4
2	1.88	1.13	0	3.27
3	1.02	1.69	0	2.58
4	0.73	2.06	0	2.28
5	0.58	2.33	0	2.12
6	0.48	2.53	0	2.00
7	0.42	2.70	0.08	1.92
8	0.37	2.85	0.14	1.86

Upper control limit for $\bar{X}$ = $UCL_{\bar{X}} = \langle\bar{X}\rangle + A_2\bar{R}$

Lower control limit for $\bar{X}$ = $LCL_{\bar{X}} = \langle\bar{X}\rangle - A_2\bar{R}$

Upper warning limit for $\bar{X}$ = $UWL_{\bar{X}} = \langle\bar{X}\rangle + (2/3)\ A_2\bar{R}$

Lower warning limit for $\bar{X}$ = $LWL_{\bar{X}} = \langle\bar{X}\rangle - (2/3)\ A_2\bar{R}$

Upper control limit for R = $UCL_R = D_4\bar{R}$

Lower control limit for R = $LCL_R = D_3\bar{R}$

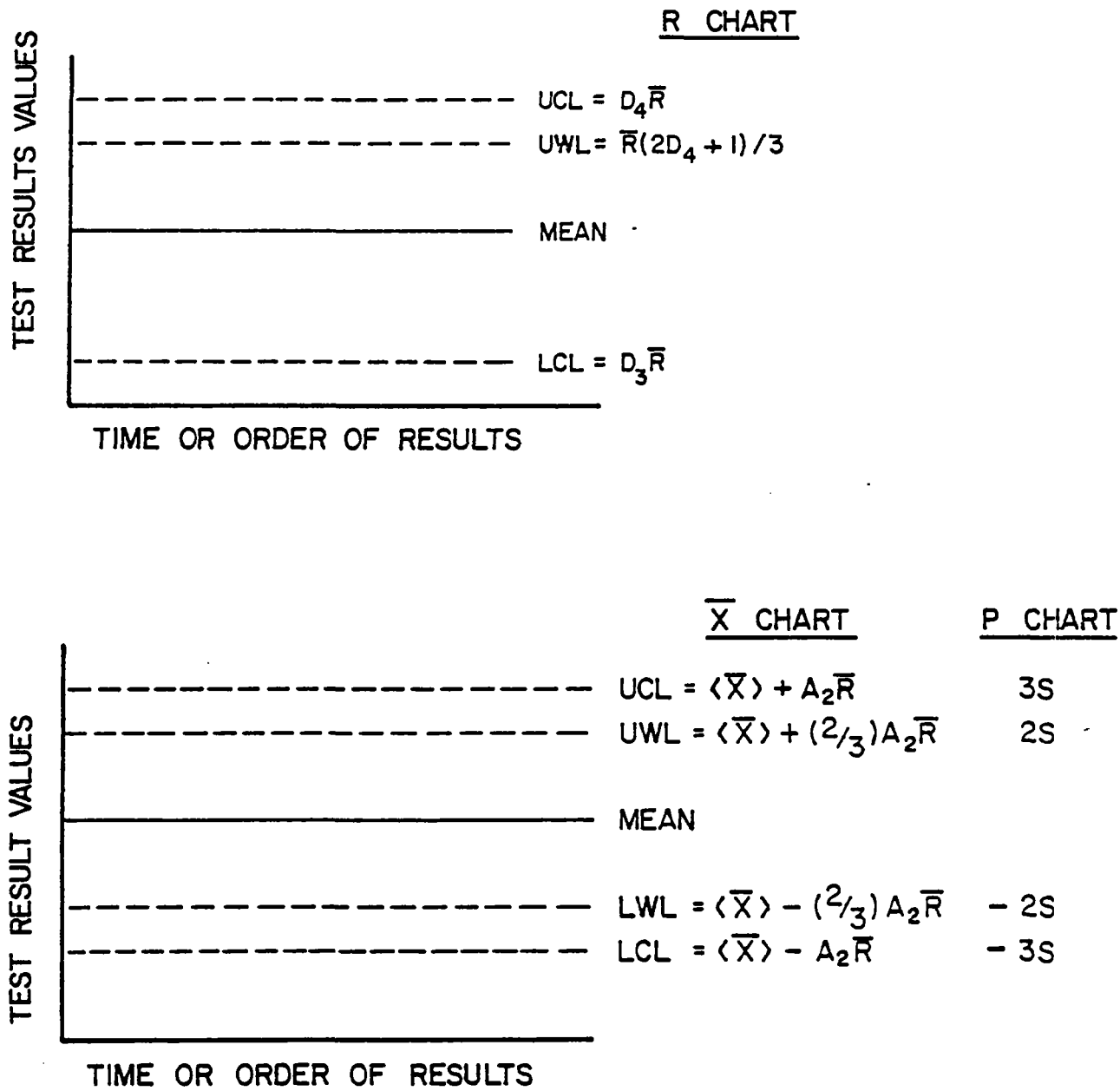
Upper Warning Limit for R = $UWL_R = \bar{R} + (2/3)(D_4\ \bar{R} - \bar{R})$

$= \bar{R}\ (2\ D_4 + 1)/3$

[1]Taken from (1) "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", March, 1979 (EPA-600/4-79-019); and (2) C. Samson, P. Hart and C. Rubin, "Fundamentals of Statistical Quality Control", Addison-Wesley (Massachusetts, 1970), p. 40.

FIGURE 3

ESSENTIALS OF CONTROL CHARTS



Statistical Calculations

The statistical techniques used in generating the data for $\bar{X}$ and R QC charts involves complex mathematics. The short cut methods for calculating the $\bar{X}$ and R limits are based on the equations listed in Table IV. The statistical factors A_2, D_3, D_4, etc. have been calculated by statisticians such that the CL limits involve a maximum risk of making an error only 0.1% to 0.3%. Thus, when the QC charts indicate that the analytical system is "out of control" 997 times out of 1,000 it is likely that something has actually gone wrong and corrective actions are needed. The factors are calculated to yield 3S limits. Examples of QC data and the statistical techniques used to calculate precision and accuracy QC charts follow.

Precision QC Charts ($\bar{X}$ and R Charts)

These charts are developed by using a minimum of 15 to 25 QC data results on duplicate analyses. Once these data have been collected over an extended period of time the warning and controlling limits on the QC charts are calculated by using the equations and statistical coefficients listed in Table IV. The procedure used follows:

(1) For each duplicate sample analysis calculate the range ($R_i = | X_2 - X_1 |$) and the average ($\bar{X}j = (X_2 + X_1)/2$) of the concentration of the duplicate set.

(2) Calculate the relative percent range (R^1_j) defined as

$$R^1_j = PRE/100 = R_i/\bar{X}_j$$

where PRE is the relative error defined in Section VIII.

(3) Calculate the mean ($\bar{R}^1$) relative range by summing the R^1_j values and divide by the total number (n) of duplicate sets, e.g.,

$$\bar{R}' = \left[\sum_{j=1}^{n} R_j\right]/n$$

(4) Calculate the grand average $<\bar{X}>$, i.e., the average of the average of n sets of duplicate averages $\bar{X}_j$ by using:

$$<\bar{X}> = \left[\sum_{j=1}^{n} \bar{X}_j\right]/n$$

(5) Calculate the warning and control limits for R and $\bar{X}$ (see Table IV) by using:

<u>For R:</u>

$$UCL = D_4\ \bar{R}^1 = 3.27\ \bar{R}^1$$

$$LCL = D_3\ \bar{R}^1 = 0$$

$$UWL = \bar{R}^1\ (2D_4 + 1)/3 = 2.51\ \bar{R}^1$$

<u>For $\bar{X}$:</u>

$$UCL = <\bar{X}> + A_2\bar{R} = <\bar{X}> + 1.88\ \bar{R}$$

$$LCL = <\bar{X}> - A_2\bar{R} = <\bar{X}> - 1.88\ \bar{R}$$

$$UWL = <\bar{X}> + (2/3)\ A_2\bar{R} = <\bar{X}> + 1.25\ \bar{R}$$

$$LWL = <\bar{X}> - (2/3)\ A_2\bar{R} = <\bar{X}> - 1.25\ \bar{R}$$

where for duplicates $D_3 = 0$, $D_4 = 3.27$, and $A_2 = 1.88$ (Table IV); UCL and LCL are the upper and lower control limits, respectively; and UWL and LWL are the upper and lower warning limits. The WL's and CL's correspond, respectively, to the 95% (2S) and 99.7% (3S) confidence limits of a normal distribution curve.

(6) Graph the $\bar{R}^1$, UCL, LCL and UWL on the QC charts with appropriate scales which allow additions of new results (Figure 3) and the individual (R^1_j) QC data results.

(7) Graph the $<\bar{X}>$, UCL, LCL, UWL, and LWL on the QC charts with appropriate scales which allow additions of new results and individual ($\bar{X}_j$) QC data.

(8) If QC values are "out of control", i.e., lie outside the control limits, take appropriate corrective action.

Accuracy QC Charts (P Charts)

The P charts are the same as the $\bar{X}$ and R charts since their function is to enable us to detect changes in the laboratory daily performance of analyses and take corrective action. The P QC charts utilize the sigma (i.e., standard deviation, S) as a quantitative measure of the degree of variations in the analytical methodologies.

The accuracy of the laboratory analytical methodologies is monitored via the analysis of various spiked samples and/or audits of synthetic standards. Spiked samples are also analyzed vis a vis field samples and the percent recovery calculated. Once a minimum of 15 QC recovery data have been collected over a period of time the warning and controlling limits are calculated and P charts developed. The procedure used follows:

(1) For each spiked sample analyzed calculate the percent recovery (PR) using the equations given in Section VIII.

(2) Calculate the mean percent recovery ($\bar{PR}$) by summing the total number of PR's and divide by n (see Section VIII).

(3) Calculate the standard deviation (S) from the percent recoveries (see Section VIII).

(4) Calculate the warning (WL) and control (CL) limits by using:

$$CL = \text{mean} \pm 3S$$

$$WL = \text{mean} \pm 2S$$

where CL and WL denote, respectively, the upper and lower control limits, and the upper and lower warning limits; S the standard deviation; and mean the average percent recovery ($\bar{PR}$) for n spiked samples or synthetic standards. The WL and CL on the accuracy charts (similar to the precision charts) correspond, respectively, to the 95% and 99.7% confidence limits of a normal distribution curve.

(5) Graph the mean, WL, CL and the individual (PR) QC data results on the accuracy chart using appropriate scales.

(6) If QC values lie outside the control limits, the analytical method is "out of control" and appropriate corrective actions are taken.

The "Daily QC Model"

The "Daily QC Model" comprises two unique activities of our QA/QC program, i.e., the data management and monitoring specific statistical programs of data management systems on a daily basis. The salient features of the programs are discussed below.

1. Data Management

Integral to the laboratory's QA/QC program is the management of data generated from specified quality control procedures. These procedures are designed to monitor all laboratory analyses and ultimately, to ensure the highest possible quality of results. As

previously mentioned, the duplicate, the spiked recovery, the synthetic known and the blank(s) are the analytical tools used to monitor the precision and accuracy of analytical methods. Recall:

(a) duplicate analyses monitor analytical method precision,

(b) spiked samples and synthetic knowns monitor analytical accuracy, and

(c) analyses of blanks account for possible sources of contamination.

The data produced from these tests is maintained via a quality control data management system which has the dual function of relating QA/QC data to analytical performance on a daily as well as varying time frames.

The key to the management of QA/QC data in the laboratory is the Firm's Honeywell X560 computer. Quality control computer programs allow for the calculations, storage, segregation, interpretation, monitoring and retrieval of each bit of QA/QC information. A discrete system of sample identification is used which allows the computer to perform these functions automatically. Each QA/QC sample is assigned a specific code identifying it as a blank, duplicate, spike or synthetic known sample. The code identifiers place each QC value in an appropriate data base which provides a permanent record of each and every quality control sample. These data base are then used as the starting point of various statistical analyses of QC data which aid in understanding the developed analytical information.

Specific statistical programs are available for the various types of QA/QC samples, and generate precision (X bar and R) and accuracy (P bar) quality control charts. These charts provide the graphic

representation of the QA/QC information and are used to monitor the accuracy and precision of the various analytical methodologies daily.

2. Monitoring Statistical Programs of Data Management Systems

The QA/QC programs are made available to the QA/QC group leader and the analyst to allow daily response to analysis. The programs offer instant presentation of statistical values which are checked vis a vis the most recent mean, standard deviations and control limits calculated from each data base in the computer. As a result the QA/QC group leader and the analyst will know immediately whether or not the analytical method performance is in control (lie within acceptable ranges) and a decision can be made to accept, reject or repeat the analysis.

In addition, a program exists for the QA/QC group leader which presents all quality control information in a daily printout (see Figures 4 and 5). On this printout, information concerning QC samples is organized for review by the QA/QC group leader. The sample number, the test parameter, the QC sample type, the date of analysis, percent recoveries, relative errors and all values necessary for the calculation of QC data are collected on this printout (Figure 4). In addition to the QC values, commensurate warning and control limits are given. The QA/QC group leader is able to examine these data for acceptability. A quick scan can tell him the status of unfinished samples and values of QC data entering data bases. It is at this point where errors are detected, researched, and corrected whenever possible. We feel that the use of this monitoring program minimizes elapsed time between analysis and data review, therefore, greatly

TABLE V. SUMMARY OF VARIOUS QA/QC ITEMS ON DAILY COMPUTER PRINTOUT

ITEM	INFORMATION
CONTROL CHARTS	X Bar and R Charts (precision) P Charts (accuracy)
TABLES	Blanks Duplicates (Percent Relative Error) Spikes (Percent Recovery) Synthetic Standards (Percent Error)
WARNING PROGRAM	Outliers on all QC Data Base Mean and Standard Deviation Upper and Lower Warning and Control Limits
STATISTICS	Average, Mean and Standard Deviation Upper and Lower Warning and Control Limits Skewness and Kurtosis Percent Relative Error Percent Recovery Percent Error

improves the sensitivity of our QC program to our analyses. The earlier errors are detected and corrected, the less time is required to deliver valid results to a client.

A summary of the various QC activities and statistical calculations found in the daily printout is given in Table V. If QC values are found to lie outside the control limits, corrective actions are taken to bring the analytical method "under control". The various corrective actions are delineated in Table VI.

3. Other QA/QC Functions

A further ramification of the QA/QC computer management system is the historical evaluations afforded through data storage. Data may be retrieved over long varying time frames providing solid estimates of performance limits for any given analytical parameter. By the same token knowledge of performance limits and the factors that establish them should allow for the improvement of analyses as these factors are identified and removed. Such review is used in the evaluation of new techniques, instruments, and analysts when comparisons are made to the established quality control data bases.

To assist in evaluation and historical review a statistical package is available for measuring the variability of any given data over varying time frames. The Peursonian coefficient of skewness is utilized to quantify variability of percent recoveries, duplicate ratios, and percent of unknown values.

Automatic storage of data, generation of control charts, and data examination through statistics are the tools used to manage the quality control data. The goal of the data management system is a sensitive quality control program which will allow accurate decision making processes and continuous quality of analytical results.

X. FOR THE CLIENT

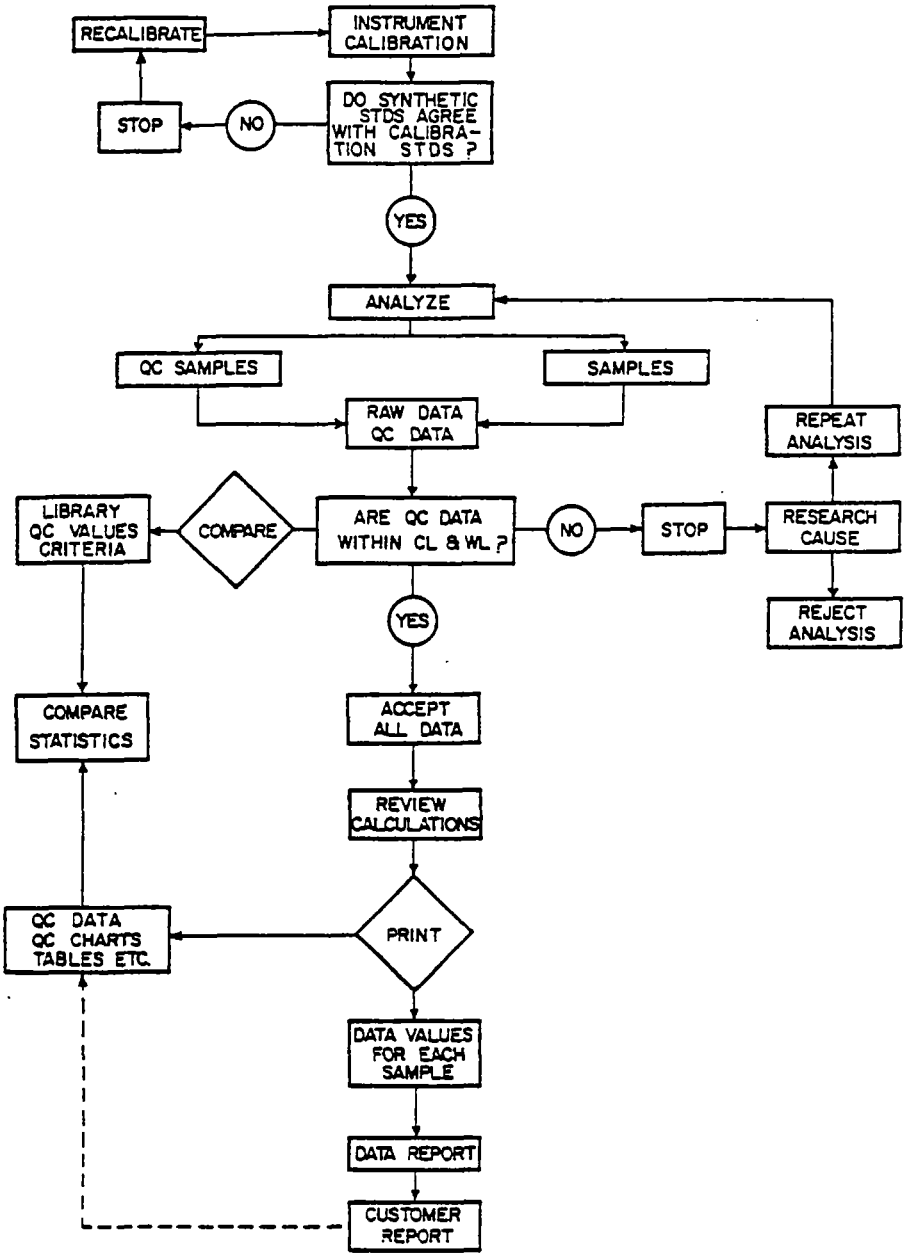
The overall importance of our quality control program to the client lies in the fact that we are able to guarantee a certain level of confidence in our analyses. This confidence is expressed through our statistics. As mentioned earlier, we have established our acceptability limits to be plus or minus three times the standard deviation of the mean of the quality control values in each data base. Assuming that the values in the data bases describe a normal distribution, it is known that 99% of the values will fall within the range described by 3 standard deviations of the mean of the distribution. There exists a probability of .99 that any data point will be (plus or minus) 3 times the standard deviation of the mean. This may be described as the 99% confidence interval. We may state, therefore, with 99% certainty, that our quality control data will fall within acceptable limits. As we use quality control data to determine the validity of analyses of client samples, the same confidence interval may be ascribed to such data. The client must be aware, however, that the limits of acceptability are based upon the actual quality control data itself. That data derived from quality control analyses directly reflects the variability of the test. The limits, therefore, will vary as the test varies. Accordingly, the confidence interval of 99% will depict a different range in concentration for each test. The use of the confidence interval provides us with a method of checking the quality of our data and providing the client with some guarantee of validity.

The other facet of our operation which must be described is the ability to adapt our quality control options to the client's specific needs. Quality control parameters, blanks, spiked samples, duplicates,

TABLE VI

DECISION MAKING PROCESS FOR QA/AC

PROTOCOL AND ANALYSIS OF SAMPLES



and the analysis of knowns may increase or decrease in frequency according to the client's wishes. If, for example, there is a concern over contamination, a client may wish to increase the number of blanks from one per ten client samples to two per set. The same applies to spikes, duplicates, and knowns.

If requested, graphs of all quality control data and lists of the statistical information can be made available. The graphs include sample numbers, mean, warning limits and control limits for acceptability (see Figures 6 and 7). The graphs may be formulated to include any desired number of data points for each of the quality control parameters. Statistical lists for data groups include the mean, standard deviation, median, coefficient of skewness and measures of kurtosis. These values can also be modified to comprise varying groups of data points. The variation is related to the time frame the client may wish to relate the data to provide the best description of the validity of analyses on his samples.

APPENDIX

KEY FOR DAILY QUALITY CONTROL REPORT

PROJECT NO: denotes client and parameters tested.

SAMPLE: denotes O'Brien & Gere sample ticket number.

MATE: client sample that was spiked or duplicated.

TYPE: Quality control sample type as:

1 - blank sample

3 - denotes duplicate

50 - chemistry spike

51 - trace organics spike

40 - EPA known concentration

QC VALUE: value obtained for QC sample as blank value, duplicate ratio, percent recoveries for spiked and known samples.

L, WARNING: lower warning limit as (-2) times the standard deviation of the mean of the last 25 samples.

U, WARNING: upper warning limit as (+2) times the standard deviation of the mean of the last 25 samples.

SIZE: number of values in data base.

COMMENTS: as written.

TABLE VII

SPIKED RECOVERIES DATA BASE

FOR GENERATING CONTROL CHARTS & STATISTICS

HG DATABASE SIZE IS 25 NUMBER OF SAMPLES ARE 54

FIELD	SAMPLE	VALUE
1	64261	109.000
2	64456	100.000
3	64362	110.000
4	64398	100.000
5	64322	100.000
6	66507	124.000
7	81628	107.000
8	19683	67.000
9	82504	112.000
10	83870	95.000
11	81075	93.000
12	90035	104.440
13	91773	124.000
14	94289	122.000
15	95283	80.000
16	92510	98.000
17	97001	82.000
18	97055	106.000
19	97253	120.000
20	97906	96.000
21	98469	98.000
22	98743	118.000
23	99151	105.000
24	2409	129.000
25	2410	103.000
26	28598	84.711
27	28833	100.000
28	38710	86.364
29	39461	73.333
30	39629	96.000
31	39653	90.625
32	21292	99.160
33	21310	125.000
34	21741	69.841
35	22011	96.061
36	88265	95.522
37	22504	131.657
38	22611	97.222
39	14622	82.759
40	26661	104.687
41	38006	80.202
42	30037	108.333
43	30035	103.448
44	35971	89.756
45	30999	95.455
46	5590	123.529
47	7507	80.851
48	7547	71.897
49	17107	109.091
50	17514	88.462
51	17521	94.667
52	17851	111.765
54	17860	32.453

BENZ DATABASE SIZE IS 25 NUMBER OF SAMPLES ARE 45

FIELD	SAMPLE	VALUE
1	29830	109.677
2	23250	87.000
3	44382	99.000
4	5593	97.500
5	43077	100.000
6	5718	120.000
7	5716	100.000
8	9646	94.500
9	9651	105.000
10	9665	92.000
11	17041	104.065
12	17062	89.000
13	17356	95.500
14	17410	83.333
15	17411	90.244
16	17585	105.000
17	17586	105.000
18	17688	105.000
19	17767	100.000
20	17774	102.000
21	17857	77.419
22	17875	87.948
23	17930	99.500
24	18104	102.000
25	18107	105.000
26	18125	88.599
27	18170	105.000
28	18234	80.645
29	18236	84.691
30	18203	93.000
31	18366	105.000
32	18464	105.000
33	18472	105.000
34	50629	91.667
35	50648	100.000
36	50789	91.667
37	50824	108.333
38	50869	100.000
39	50867	100.000
40	50942	100.000
41	50943	105.000
42	51035	105.000
43	51049	100.000
44	51051	105.000
45	51079	91.667

TIME	SAMPLE	VALUE
1	[illegible]	101,000
2	[illegible]	107,000
3	[illegible]	111,000
4	[illegible]	100,000
5	[illegible]	116,000
6	[illegible]	113,000
7	[illegible]	113,000
8	[illegible]	107,000
9	[illegible]	109,000
10	[illegible]	98,000
11	[illegible]	120,000
12	[illegible]	98,000
13	[illegible]	117,000
14	[illegible]	106,000
15	[illegible]	118,000
16	[illegible]	132,000
17	[illegible]	104,000
18	[illegible]	111,000
19	[illegible]	127,000
20	[illegible]	115,000
21	[illegible]	107,000
22	[illegible]	102,000
23	[illegible]	104,000
24	[illegible]	102,000
25	[illegible]	126,000
26	[illegible]	104,000
27	[illegible]	112,000
28	[illegible]	105,000
29	[illegible]	120,000
30	[illegible]	101,000
31	[illegible]	105,000
32	[illegible]	120,000
33	[illegible]	114,000
34	[illegible]	107,000
35	[illegible]	110,000
36	[illegible]	101,000
37	[illegible]	82,000
38	[illegible]	105,000
39	[illegible]	104,000
40	[illegible]	109,000
41	[illegible]	107,000
42	[illegible]	101,000
43	[illegible]	100,000
44	[illegible]	111,000
45	[illegible]	111,000

TIME	SAMPLE	VALUE
46	[illegible]	102,000
47	[illegible]	101,000
48	[illegible]	94,000
49	[illegible]	101,000
50	[illegible]	103,000
51	[illegible]	101,000
52	[illegible]	98,000
53	[illegible]	99,000
54	[illegible]	101,000
55	[illegible]	99,000
56	[illegible]	99,000
57	[illegible]	98,000
58	[illegible]	96,000
59	[illegible]	98,000
60	[illegible]	96,000
61	[illegible]	97,000
62	[illegible]	97,000
63	[illegible]	95,000
64	[illegible]	99,000
65	[illegible]	97,000
66	[illegible]	96,000
67	[illegible]	96,000
68	[illegible]	96,000
69	[illegible]	95,000
70	[illegible]	94,000
71	[illegible]	95,000
72	[illegible]	94,000
73	[illegible]	95,000
74	[illegible]	95,000
75	[illegible]	94,000
76	[illegible]	95,000
77	[illegible]	95,000
78	[illegible]	95,000
79	[illegible]	95,000
80	[illegible]	96,000
81	[illegible]	98,000
82	[illegible]	96,000
83	[illegible]	96,000
84	[illegible]	97,000
85	[illegible]	96,000
86	[illegible]	95,000
87	[illegible]	96,000
88	[illegible]	96,000
89	[illegible]	95,000
90	[illegible]	97,000
91	[illegible]	98,000
92	[illegible]	97,000
93	[illegible]	97,000
94	[illegible]	97,000
95	[illegible]	97,000
96	[illegible]	98,000
97	[illegible]	98,000
98	[illegible]	98,000
99	[illegible]	98,000
100	[illegible]	98,000
101	[illegible]	98,000
102	[illegible]	98,000
103	[illegible]	98,000
104	[illegible]	98,000

TIME	SAMPLE	VALUE
105	93814	107,000
106	93850	124,000
107	91324	97,000
108	93250	122,000
109	2412	77,000
110	93383	115,000
111	93880	113,000
112	2307	86,000
113	2413	86,000
114	2409	136,000
115	99339	114,000
116	28104	95,238
117	28277	80,000
118	28369	97,561
119	28487	115,789
120	28633	99,598
121	28847	102,915
122	38623	97,872
123	38864	99,759
124	38865	95,029
125	37186	105,556
126	37268	112,195
127	39530	101,124
128	39870	100,000
129	39959	102,362
130	21162	103,226
131	21515	95,652
132	21641	91,228
133	21857	106,852
134	88134	106,977
135	88258	104,504
136	88299	90,411
137	88464	102,532
138	88506	101,961
139	88762	102,071
140	22351	103,235
141	22429	106,456
142	22630	106,667
143	11031	100,000
144	11211	100,000
145	40851	86,531
146	40856	87,640
147	40849	96,141
148	40838	92,308
149	40847	92,453
150	40844	100,000
151	40845	98,630
152	40841	100,000
153	40843	105,128
154	40842	100,000
155	40841	101,235
156	40838	105,977
157	40837	107,143

TIME	SAMPLE	VALUE
158	40838	100,000
159	43037	113,500
160	40836	107,143
161	40835	105,882
162	40833	113,500
163	40835	103,158
164	40832	102,564
165	40831	94,737
166	40830	105,747
167	40829	102,703
168	40828	105,263
169	40827	103,125
170	36073	28,562
171	37917	97,500
172	37969	100,813
173	40283	98,592
174	31498	102,703
175	67826	95,238
176	31033	98,893
177	65425	101,266
178	35804	96,774
179	7571	96,925
180	9636	104,167
181	9637	92,398
182	17061	101,031
183	17326	103,704
184	17541	107,692
185	17569	80,000
186	95674	118,000
187	99663	111,000
188	50601	100,000
189	50464	114,286
190	51103	100,000

FIGURE 4

DAILY QUALITY CONTROL REPORT

(SEE KEY)

QC REPORT FOR 3/15/83 TO 3/22/83

PROJECT NO.	SAMPLE	MATE	TYPE	DATE	QC VALUE	L. WARNING	U. WARNING	SIZE	COMMENTS
1042- 97-510	18465	0	1	3/15/83					
1042- 97-510	50585	0	1	3/16/83					
1042- 97-510	50714	0	1	3/15/83					
1042- 97-510	50783	0	1	3/15/83					
1042- 97-510	50787	0	1	3/15/83					
1042- 97-510	50790	0	1	3/16/83					
1042- 97-510	50793	0	1	3/16/83					NO SCHEDULED ANALYSES
1042- 97-510	50795	0	1	3/16/83					
1042- 97-510	50820	0	1	3/16/83					NO SCHEDULED ANALYSES
1042- 97-510	50840	0	1	3/16/83					NO SCHEDULED ANALYSES
1042- 97-510	50850	0	1	3/16/83					
1042- 97-510	50851	0	1	3/17/83					
1042- 97-510	50853	0	1	3/17/83					
1042- 97-510	50866	0	1	3/18/83					
1042- 97-510	50868	0	1	3/18/83					
1042- 97-510	50883	0	1	3/21/83					NO SCHEDULED ANALYSES
1042- 97-510	50890	0	1	3/21/83					
1042- 97-510	50891	0	1	3/21/83					
1042- 97-510	50892	0	1	3/21/83					
1042- 97-510	50897	0	1	3/21/83					NO SCHEDULED ANALYSES
1042- 97-510	50899	0	1	3/21/83					
1042- 97-510	50900	0	1	3/21/83					
1042- 97-510	50906	0	1	3/22/83					
BENZ	<1.				1.000			481	
CHCL3	<1.000				1.000			191	
FREON113	<1.000				1.000			593	
CL3CCH3	<1.000				1.000			608	
CCL4	<1.000				1.000			73	
BRCL2CH	<1.000				1.000			191	
CL3C2H	<1.000				1.000			609	
CLBR2CH	<1.000				1.000			191	
CHBR3	<10.000				10.000			190	
CL4C2	<1.000				1.000			603	
CH2CL2	<1.000				1.000			604	
TOLUENE	<1.000				1.000			482	
M-XYLENE	<1.000				1.000			473	
CLOROBZ	<1.000				1.000			171	
CL4C2H2	<1.000				1.000			66	
DCETANE	<1.000				1.000			542	

TAN12	<1.000				1.000	542
DCEN11	<1.000				1.000	542
ETHBENZ	<1.000				1.000	98
DCLEN12	<1.000				1.000	542
C2H5CL	<1.000				1.000	316
CH2CHCL	<1.000				1.000	315
XYLENES	<1.000				1.000	195
CH3CL	<1.000				1.000	66
CH3BR	<1.000				1.000	66
DCPAN12	<1.000				1.000	66
DCPENT13	<1.000				1.000	66
CL3C2112	<1.000				1.000	66
DCPENC13	<1.000				1.000	66
CLETHER	<10.000				10.000	66
1042- 97-510	50907	0	1	3/22/83		
BENZ	2.				2.000	482
CHCL3	<1.000				1.000	192
FREON113	<1.000				1.000	594
CL3CCH3	<1.000				1.000	609
CCL4	<1.000				1.000	74
BRCL2CH	<1.000				1.000	192
CL3C2H	<1.000				1.000	610
CLBR2CH	<1.000				1.000	192
CHBR3	<10.000				10.000	191
CL4C2	<1.000				1.000	604
CH2CL2	<1.000				1.000	605
TOLUENE	<1.000				1.000	483
M-XYLENE	<1.000				1.000	474
CLOROBZ	<1.000				1.000	172
CL4C2H2	<1.000				1.000	67
DCETAN11	<1.000				1.000	343
DCETAN12	<1.000				1.000	343
DCLEN11	<1.000				1.000	343
ETHBENZ	<1.000				1.000	99
DCLEN12	<1.000				1.000	343
C2H5CL	<1.000				1.000	317
CH2CHCL	<1.000				1.000	316
XYLENES	<1.000				1.000	196
CH3CL	<1.000				1.000	67
CH3BR	<1.000				1.000	67
DCPAN12	<1.000				1.000	67
DCPENT13	<1.000				1.000	67
CL3C2112	<1.000				1.000	67
DCPENC13	<1.000				1.000	67
CLETHER	<10.000				10.000	67
1042- 97-510	50913	0	1	3/22/83		NO SCHEDULED ANALYSES
1042- 97-510	50700	468	3	3/16/83		
CHCL3	<100.000					< FLAG - SKIPPED
FREON113	<10.000					< FLAG - SKIPPED
CCL4	<100.000					< FLAG - SKIPPED
BRCL2CH	<100.000					< FLAG - SKIPPED
CLBR2CH	<10.000					< FLAG - SKIPPED
CHBR3	<100.000					< FLAG - SKIPPED
CL4C2	<10.000					< FLAG - SKIPPED
CLOROBZ	<1.000					< FLAG - SKIPPED
CL4C2H2	<10.000					< FLAG - SKIPPED
CH3CL	<10.000					< FLAG - SKIPPED
CH3BR	<10.000					< FLAG - SKIPPED
DCPAN12	<10.000					< FLAG - SKIPPED
DCPENT13	<10.000					< FLAG - SKIPPED
CL3C2112	<10.000					< FLAG - SKIPPED

D NC13	<10,000				< FLAG - SKIPPED
CLETHER	<100,000				< FLAG - SKIPPED
1042- 97-510	50741	50686	3	3/16/83	
BOD5	<10.0				< FLAG - SKIPPED
1042- 97-510	50791	50605	3	3/16/83	
1042- 97-510	50817	50812	3	3/16/83	NO SCHEDULED ANALYSES
1042- 97-510	50818	50660	3	3/16/83	
1042- 97-510	50821	50660	3	3/16/83	
NO2N	<.01				< FLAG - SKIPPED
1042- 97-510	50823	493	3	3/16/83	
BENZ	<10.				< FLAG - SKIPPED
CHCL3	<100,000				< FLAG - SKIPPED
FREON113	<100,000				< FLAG - SKIPPED
CCL4	<1000,000				< FLAG - SKIPPED
BRCL2CH	<1000,000				< FLAG - SKIPPED
CLBR2CH	<100,000				< FLAG - SKIPPED
CHBR3	<1000,000				< FLAG - SKIPPED
CLOROBZ	<10,000				< FLAG - SKIPPED
CL4C2H2	<100,000				< FLAG - SKIPPED
ETHBENZ	<10,000				< FLAG - SKIPPED
C2H5CL	<100,000				< FLAG - SKIPPED
CH2CHCL	<100,000				< FLAG - SKIPPED
CH3CL	<100,000				< FLAG - SKIPPED
CH3BR	<100,000				< FLAG - SKIPPED
DCPAN12	<100,000				< FLAG - SKIPPED
DCPENT13	<100,000				< FLAG - SKIPPED
CL3C2H2	<100,000				< FLAG - SKIPPED
DCPENC13	<100,000				< FLAG - SKIPPED
CLETHER	<1000,000				< FLAG - SKIPPED
1042- 97-510	50825	212	3	3/16/83	
1042- 97-510	50827	212	3	3/16/83	
NO2N	<.01				< FLAG - SKIPPED
1042- 97-510	50849	511	3	3/16/83	
BENZ	<100.				< FLAG - SKIPPED
CHCL3	<1000,000				< FLAG - SKIPPED
FREON113	<1000,000				< FLAG - SKIPPED
CCL4	<1000,000				< FLAG - SKIPPED
BRCL2CH	<1000,000				< FLAG - SKIPPED
CLBR2CH	<1000,000				< FLAG - SKIPPED
CHBR3	<10000,000				< FLAG - SKIPPED
CL4C2H2	<1000,000				< FLAG - SKIPPED
DCETAN11	<1000,000				< FLAG - SKIPPED
DCETAN12	<1000,000				< FLAG - SKIPPED
ETHBENZ	<100,000				< FLAG - SKIPPED
C2H5CL	<1000,000				< FLAG - SKIPPED
CH2CHCL	<1000,000				< FLAG - SKIPPED
CH3CL	<1000,000				< FLAG - SKIPPED
CH3BR	<1000,000				< FLAG - SKIPPED
DCPAN12	<1000,000				< FLAG - SKIPPED
DCPENT13	<1000,000				< FLAG - SKIPPED
CL3C2H2	<1000,000				< FLAG - SKIPPED
DCPENC13	<1000,000				< FLAG - SKIPPED
CLETHER	<10000,000				< FLAG - SKIPPED
1042- 97-510	50852	3892	3	3/17/83	
BENZ	<1.				< FLAG - SKIPPED

	CL3	<1.000			< FLAG - SKIPPED
	FREON113	<1.000			< FLAG - SKIPPED
	CCL4	<1.000			< FLAG - SKIPPED
	BRCL2CH	<1.000			< FLAG - SKIPPED
	CLBR2CH	<1.000			< FLAG - SKIPPED
	CHBR3	<10.000			< FLAG - SKIPPED
	CL4C2	<1.000			< FLAG - SKIPPED
	TOLUENE	<1.000			< FLAG - SKIPPED
	M-XYLENE	<1.000			< FLAG - SKIPPED
	CLOROBZ	<1.000			< FLAG - SKIPPED
	CL4C2H2	<1.000			< FLAG - SKIPPED
	ETHBENZ	<1.000			< FLAG - SKIPPED
	XYLENES	<1.000			< FLAG - SKIPPED
	CH3BR	<1.000			< FLAG - SKIPPED
	DCPAN12	<1.000			< FLAG - SKIPPED
	DCPENT13	<1.000			< FLAG - SKIPPED
	CL3C2112	<1.000			< FLAG - SKIPPED
	DCPENC13	<1.000			< FLAG - SKIPPED
	CLETHER	<10.000			< FLAG - SKIPPED
1042- 97-510		50854	3858	3	3/18/83
	CHCL3	<1.000			< FLAG - SKIPPED
	CCL4	<1.000			< FLAG - SKIPPED
	BRCL2CH	<1.000			< FLAG - SKIPPED
	CLBR2CH	<1.000			< FLAG - SKIPPED
	CHBR3	<10.000			< FLAG - SKIPPED
	TOLUENE	<1.000			< FLAG - SKIPPED
	M-XYLENE	<1.000			< FLAG - SKIPPED
	CLOROBZ	<1.000			< FLAG - SKIPPED
	CL4C2H2	<1.000			< FLAG - SKIPPED
	DCETAN12	<1.000			< FLAG - SKIPPED
	CH3CL	<10.000			< FLAG - SKIPPED
	CH3BR	<10.000			< FLAG - SKIPPED
	DCPAN12	<1.000			< FLAG - SKIPPED
	DCPENT13	<1.000			< FLAG - SKIPPED
	CL3C2112	<1.000			< FLAG - SKIPPED
	DCPENC13	<1.000			< FLAG - SKIPPED
	CLETHER	<10.000			< FLAG - SKIPPED
1042- 97-510		50856	18315	3	3/16/83
	AL	<.1			< FLAG - SKIPPED
1042- 97-510		50858	215	3	3/17/83 NO SCHEDULED ANALYSES
1042- 97-510		50860	50660	3	3/17/83
	F	<.10			< FLAG - SKIPPED
1042- 97-510		50862	18338	3	3/17/83
1042- 97-510		50885	212	3	3/21/83
1042- 97-510		50898	3902	3	3/21/83
	BENZ	<100.			< FLAG - SKIPPED
	CHCL3	<1000.000			< FLAG - SKIPPED
	FREON113	<1000.000			< FLAG - SKIPPED
	CCL4	<1000.000			< FLAG - SKIPPED
	BRCL2CH	<1000.000			< FLAG - SKIPPED
	CLBR2CH	<1000.000			< FLAG - SKIPPED
	CHBR3	<10000.000			< FLAG - SKIPPED
	CL4C2	<1000.000			< FLAG - SKIPPED
	CH2CL2	<1000.000			< FLAG - SKIPPED
	TOLUENE	<100.000			< FLAG - SKIPPED
	M-XYLENE	<100.000			< FLAG - SKIPPED
	CLOROBZ	<100.000			< FLAG - SKIPPED
	CL4C2H2	<1000.000			< FLAG - SKIPPED

TAN12	<1000.000								< FLAG - SKIPPED
LEN11	<1000.000								< FLAG - SKIPPED
ETHBENZ	<100.000								< FLAG - SKIPPED
C2H5CL	<1000.000								< FLAG - SKIPPED
CH2CHCL	<1000.000								< FLAG - SKIPPED
XYLENES	<100.000								< FLAG - SKIPPED
CH3CL	<1000.000								< FLAG - SKIPPED
CH3BR	<1000.000								< FLAG - SKIPPED
DCPAN12	<1000.000								< FLAG - SKIPPED
DCPENT13	<1000.000								< FLAG - SKIPPED
CL3C2112	<1000.000								< FLAG - SKIPPED
DCPENC13	<1000.000								< FLAG - SKIPPED
CLETHER	<10000.000								< FLAG - SKIPPED
1042- 97-510	50901	50800	3	3/21/83					
BENZ	<1.								< FLAG - SKIPPED
CHCL3	<1.000								< FLAG - SKIPPED
CCL4	<10.000								< FLAG - SKIPPED
BRCL2CH	<10.000								< FLAG - SKIPPED
CLBR2CH	<1.000								< FLAG - SKIPPED
CHBR3	<10.000								< FLAG - SKIPPED
M-XYLENE	<1.000								< FLAG - SKIPPED
CLOROBZ	<1.000								< FLAG - SKIPPED
CL4C2H2	<1.000								< FLAG - SKIPPED
DCETAN12	<1.000								< FLAG - SKIPPED
ETHBENZ	<1.000								< FLAG - SKIPPED
DCLEN12	<1.000								< FLAG - SKIPPED
C2H5CL	<1.000								< FLAG - SKIPPED
CH2CHCL	<1.000								< FLAG - SKIPPED
XYLENES	<1.000								< FLAG - SKIPPED
CH3CL	<1.000								< FLAG - SKIPPED
CH3BR	<1.000								< FLAG - SKIPPED
DCPAN12	<1.000								< FLAG - SKIPPED
DCPENT13	<1.000								< FLAG - SKIPPED
CL3C2112	<1.000								< FLAG - SKIPPED
DCPENC13	<1.000								< FLAG - SKIPPED
CLETHER	<10.000								< FLAG - SKIPPED
1042- 97-510	50902	18543	3	3/21/83					NO SCHEDULED ANALYSES
1042- 97-510	50909	212	3	3/22/83					
NO2NO3	.45	.41			1.103	.8154	1.3219	36	
1042- 97-510	50911	212	3	3/22/83					
NO2N	<.01								< FLAG - SKIPPED
1042- 97-510	50916	213	3	3/22/83					
ZN	.33	.25			1.320 *	.9379	1.1329	49	
1042- 97-510	50918	219	3	3/22/83					
CU	.24	.24			1.000	.9394	1.1100	45	
1042- 97-510	50788	50807	40	3/18/83					
1042- 97-510	50789	50629	40	3/16/83					
1042- 97-510	50807	0	40	3/16/83					
1042- 97-510	50824	50629	40	3/16/83					
1042- 97-510	50867	50869	40	3/22/83					
BENZ	2.	2.			100.000	78.6607	115.0226	39	
BENZ,F	2.								
CL3CCH3	2.000	2.000			100.000	71.9670	102.8464	39	
CL3CCH3,F	2.000								

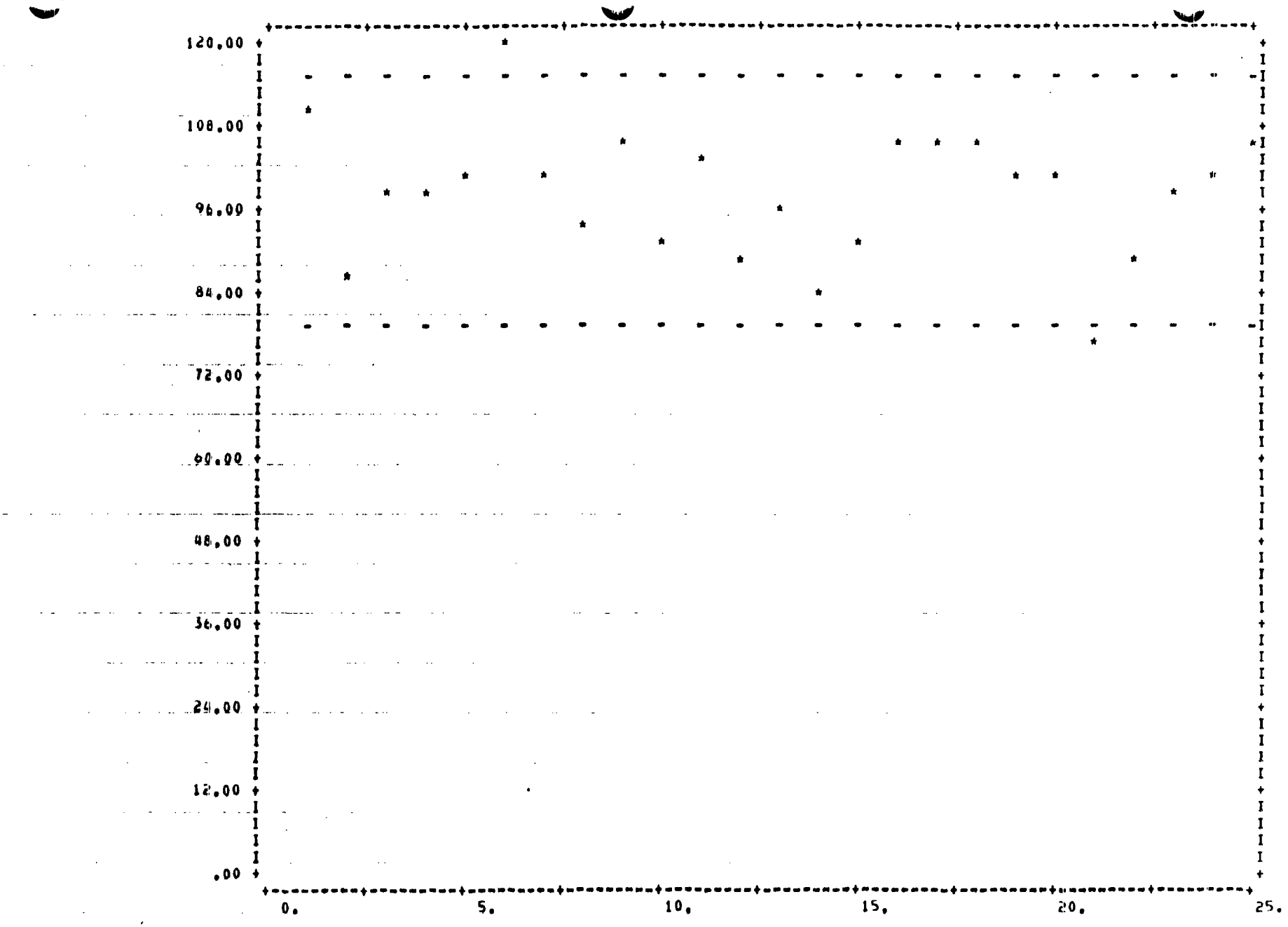
TC2H	2.000	2.000		100.000	80.0254	114.7254	39	
TC2H,F	2.000							
CL4C2	2.000			100.000	.0000	.0000	15	
CL4C2,F	2.000							
CH2CL2	2.000	2.000		100.000	75.9820	105.6501	31	
CH2CL2,F	2.000							
TOLUENE	2.000	2.000		100.000	77.4026	115.4676	39	
TOLUENE,F	2.000							
CLOROBZ	2.000	2.000		100.000	48.9895	128.4501	25	
CLOROBZ,F	2.000							
DCETAN11	2.000	2.000		100.000 *	78.4276	96.3720	27	
DCETAN11,F	2.000							
DCETAN12	2.000	2.000		100.000	52.9445	105.6725	33	
DCETAN12,F	2.000							
DCLEN11	2.000	2.000		100.000	69.3339	110.3648	33	
DCLEN11,F	2.000							
ETHBENZ	2.000	2.000		100.000	79.7258	107.1142	37	
ETHBENZ,F	2.000							
DCLEN12	2.000	2.000		100.000	73.1254	102.0690	31	
DCLEN12,F	2.000							
1042- 97-510	50869	0	40	3/18/83				
1042- 97-510	50645	50605	50	3/16/83				
1042- 97-510	50667	17868	50	3/15/83				
TCOLI	0.							QC FILT/UNFILT ANALYSIS NOT DONE
1042- 97-510	50739	50650	50	3/16/83				
1042- 97-510	50742	50686	50	3/16/83				
1042- 97-510	50792	50604	50	3/16/83				
1042- 97-510	50819	50660	50	3/16/83				
1042- 97-510	50822	50660	50	3/16/83				
1042- 97-510	50826	212	50	3/16/83				
1042- 97-510	50828	212	50	3/16/83				
1042- 97-510	50855	18173	50	3/16/83				
1042- 97-510	50857	214	50	3/17/83				
1042- 97-510	50859	18268	50	3/17/83				
1042- 97-510	50861	50660	50	3/17/83				
1042- 97-510	50863	18338	50	3/17/83				
1042- 97-510	50893	213	50	3/21/83				
TC	61.							QC FILT/UNFILT ANALYSIS NOT DONE
TIC	52.							QC FILT/UNFILT ANALYSIS NOT DONE
TOC	9.							QC FILT/UNFILT ANALYSIS NOT DONE
1042- 97-510	50903	240	50	3/21/83				NO SCHEDULED ANALYSES
1042- 97-510	50904	18333	50	3/22/83				
HG	1.0	.5			78.947	63.5830	128.5509	54

	2.0								
1042- 97-510	50910	212	50	3/22/83					
NO2N03	.52	1.04			71.943	71.6096	136.7643	112	
NO2NO3,F	.41								
1042- 97-510	50912	212	50	3/22/83					
NO2N	.09	<.10			100.000	79.3854	132.8427	96	
NO2N,F	.19								
1042- 97-510	50915	218	50	3/22/83					
ZN	.62	.20			103.333	91.8367	117.0568	156	
ZN,F	1.00								
1042- 97-510	50917	267	50	3/22/83					
CU	.67	.35			99.259	92.2782	108.3764	149	
CU,F	1.00								
1042- 97-510	18466	18465	51	3/15/83					
1042- 97-510	50794	50793	51	3/16/83					NO SCHEDULED ANALYSES
1042- 97-510	50816	50815	51	3/16/83					NO SCHEDULED ANALYSES
1042- 97-510	50884	230	51	3/21/83					NO SCHEDULED ANALYSES
1042- 97-510	50908	50907	51	3/22/83					
BENZ	36.	2.			100.000	70.5872	120.1899	143	
BENZ,F	36.								
TOLUENE	35.000	<1.000			97.222	82.1822	110.5127	148	
TOLUENE,F	36.000								
M-XYLENE	41.000	<1.000			105.128	81.6048	111.9041	135	
M-XYLENE,F	39.000								
CLOROBZ	41.000	<1.000			95.349	57.8437	128.8618	31	
CLOROBZ,F	43.000								
ETHBENZ	41.000	<1.000			100.000	.0000	.0000	7	
ETHBENZ,F	41.000								

FIGURE 5

DAILY QUALITY CONTROL GRAPH

(SPIKED RECOVERIES)



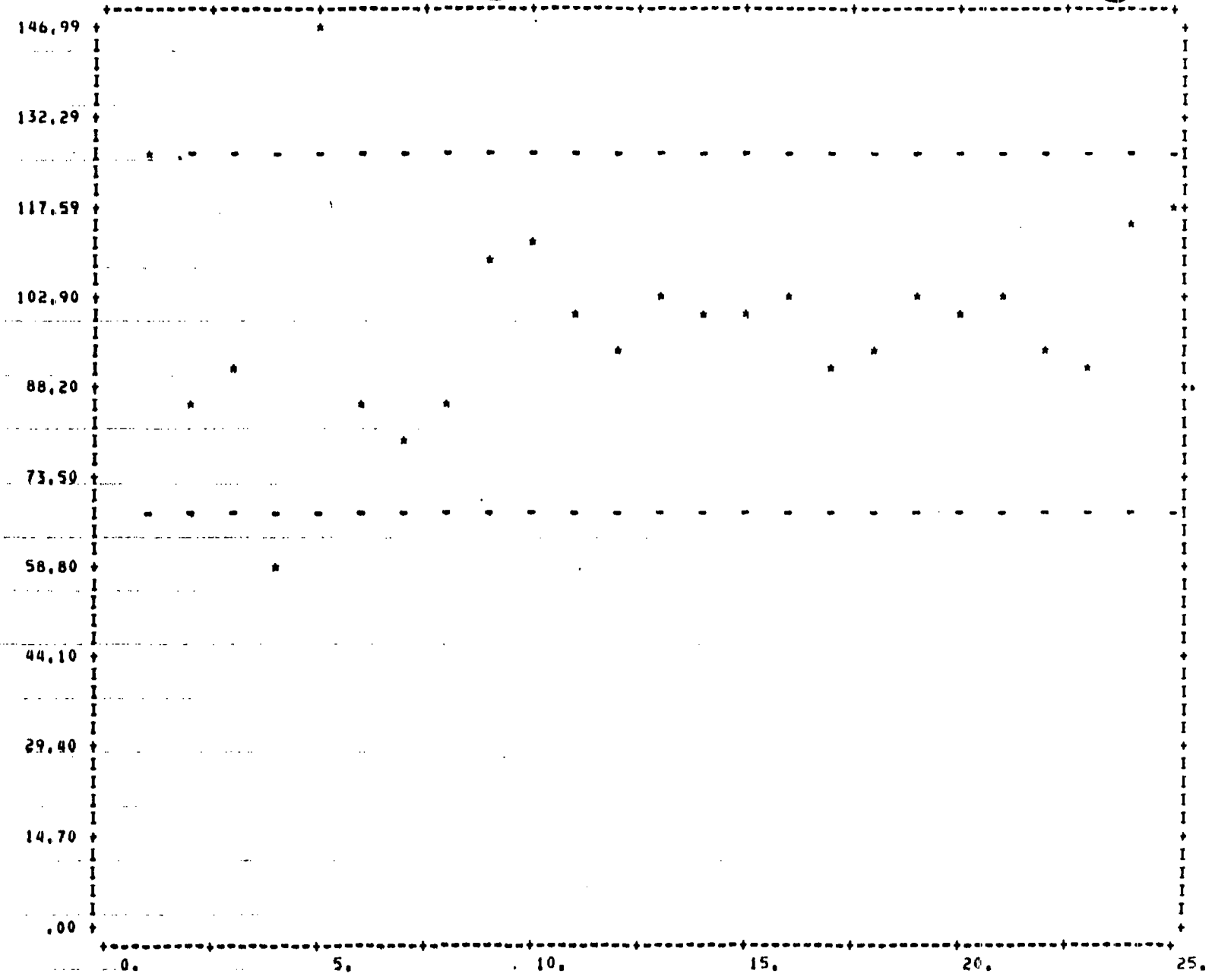
BENZ SAMPLES 29830 THROUGH 50867

(*) = QC VALUES (-) =WARNING LIMITS

MEAN = 97.8021 STANDARD DEVIATION = 8.87217

MEDIAN = 100.000 SKEWNESS = -.749181 KURTOSIS = 2.95627

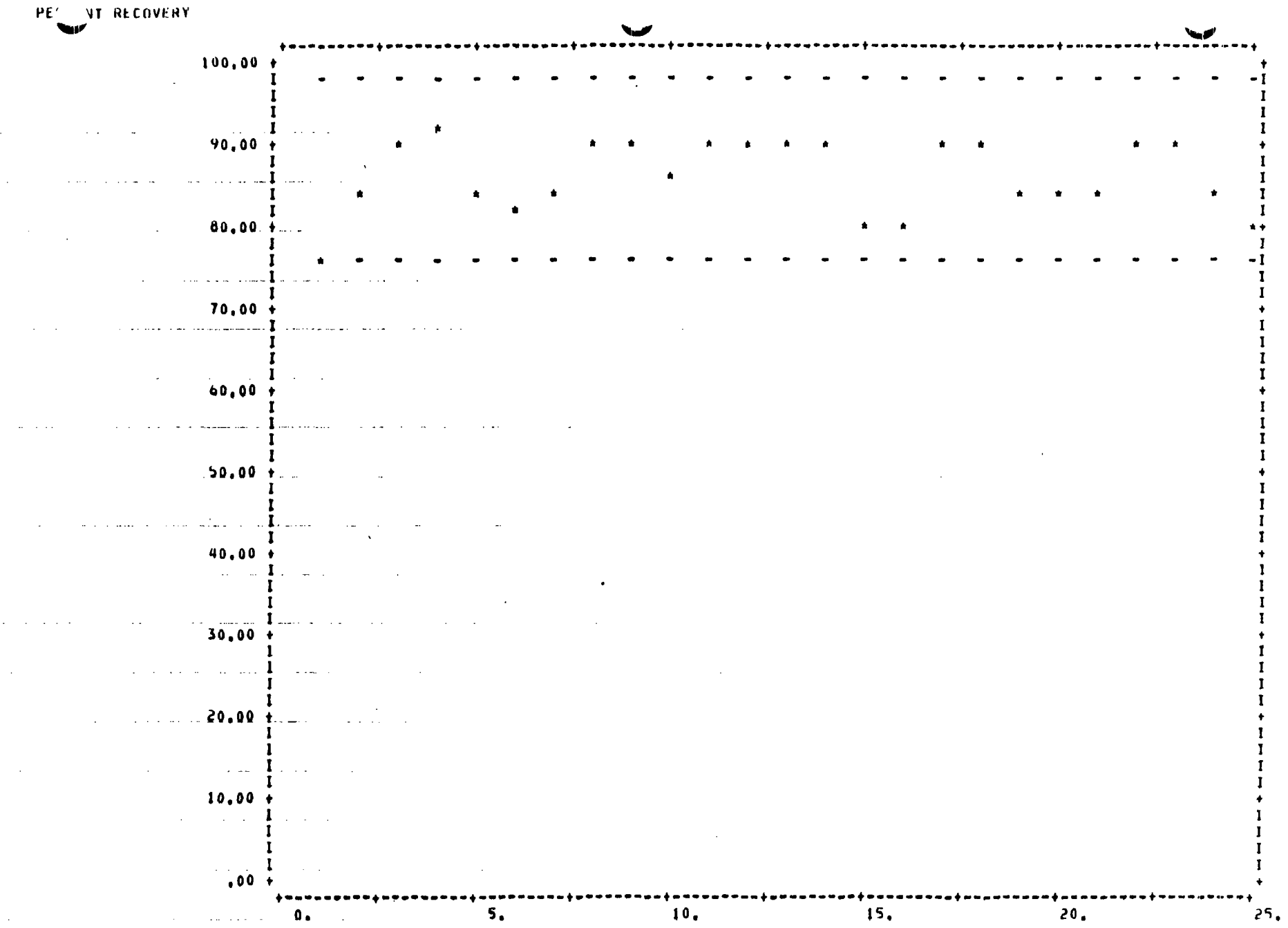
PE' NT RECOVERY



CL3C2H SAMPLES 39705 THROUGH 50867

(*) - QC VALUES (-) -WARNING LIMITS

MEAN = 97.7033 STANDARD DEVIATION = 14.3505
MEDIAN = 100.000 SKEWNESS = -.480136 KURTOSIS = 5.97265



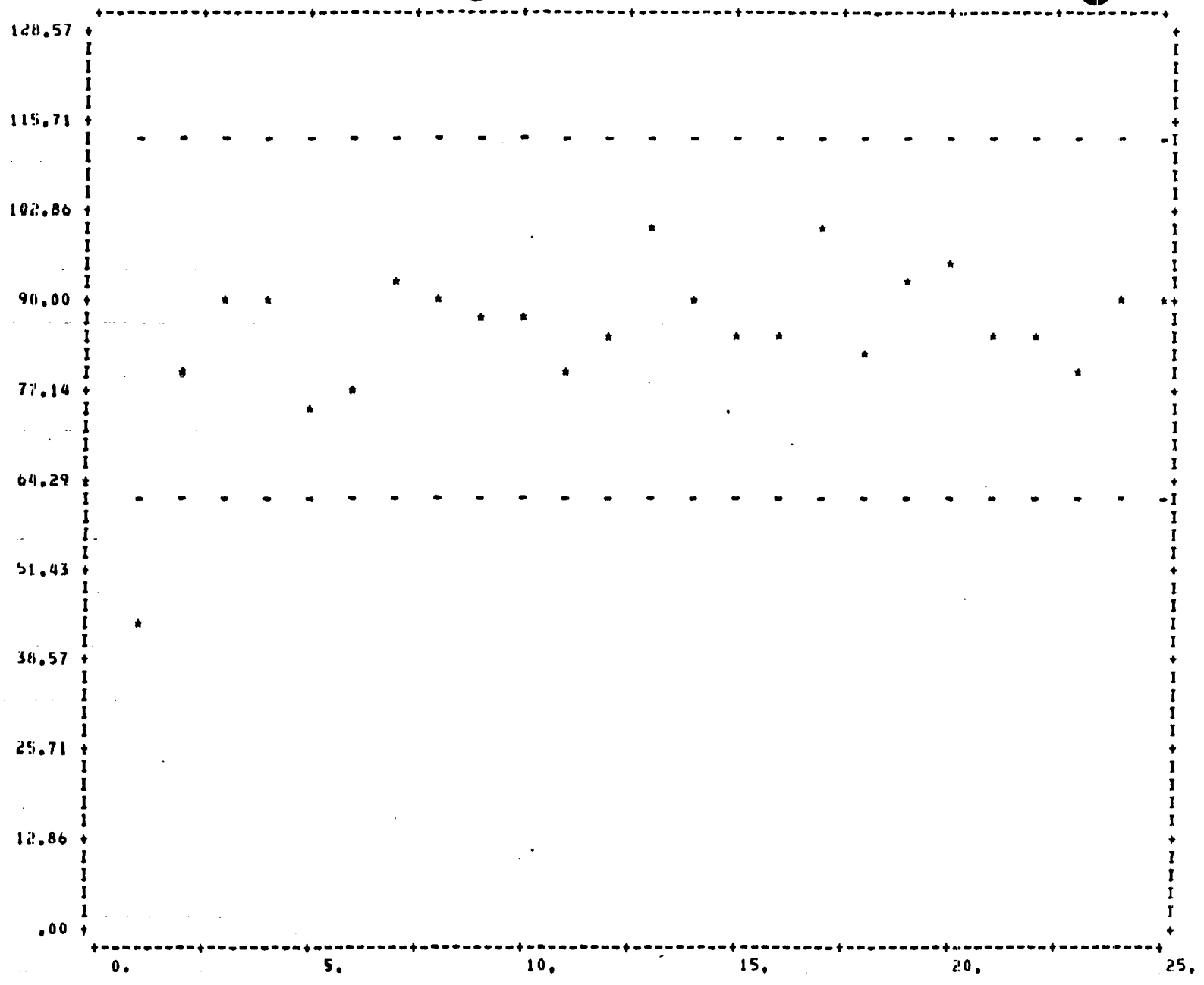
DCETAN11 SAMPLES 23250 THROUGH 50867

(*) - QC VALUES (-) -WARNING LIMITS

MEAN = 87.4443 STANDARD DEVIATION = 5.44788

MEDIAN = 89.0000 SKEWNESS = -.858704 KURTOSIS = 3.53358

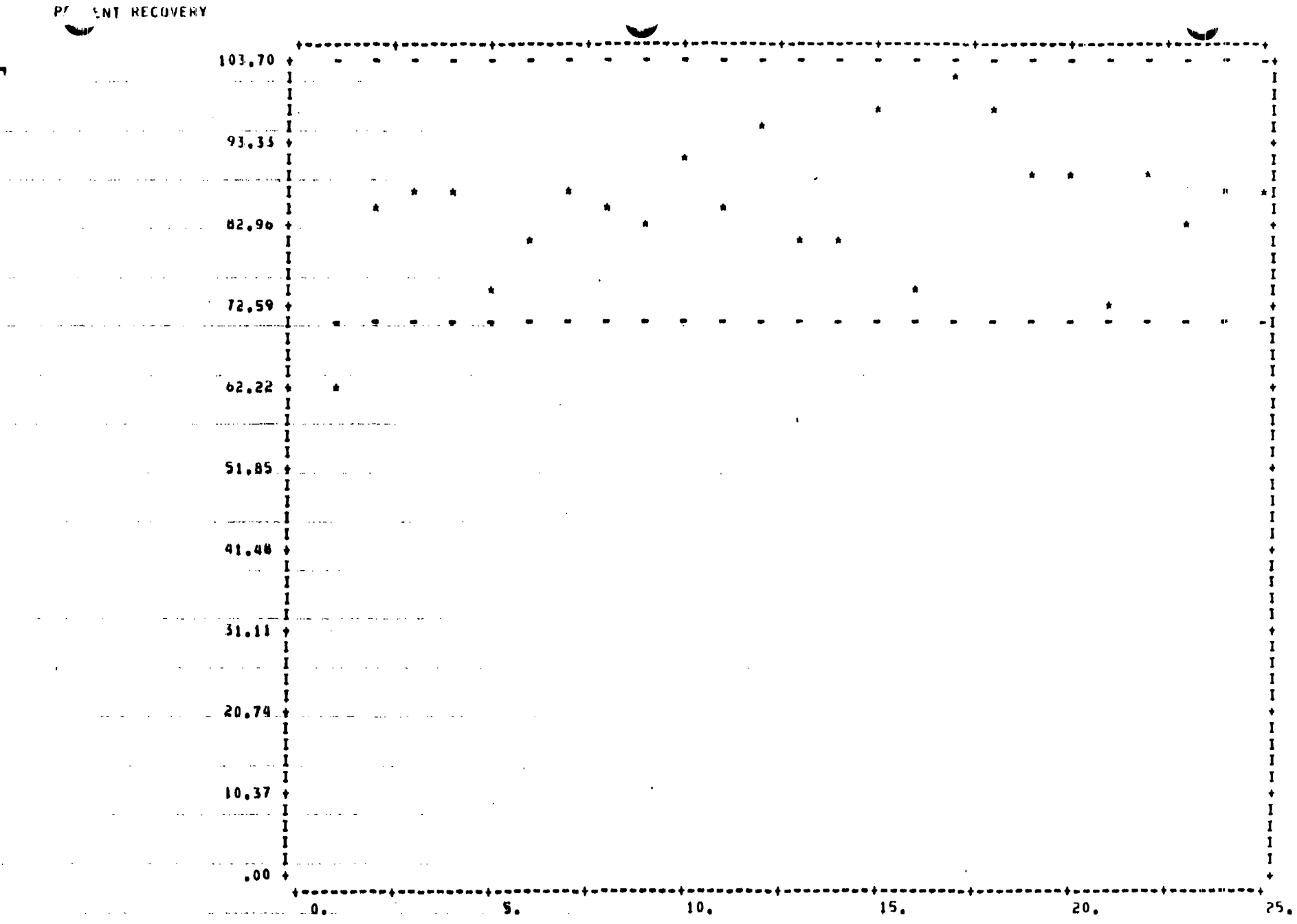
P ENT RECOVERY



DCLEN11 SAMPLES 23250 THROUGH 50867

(*) - QC VALUES (-) -WARNING LIMITS

MEAN = 87.7040 STANDARD DEVIATION = 12.5846
MEDIAN = 88.5000 SKEWNESS = -.189742 KURTOSIS = 8.48701



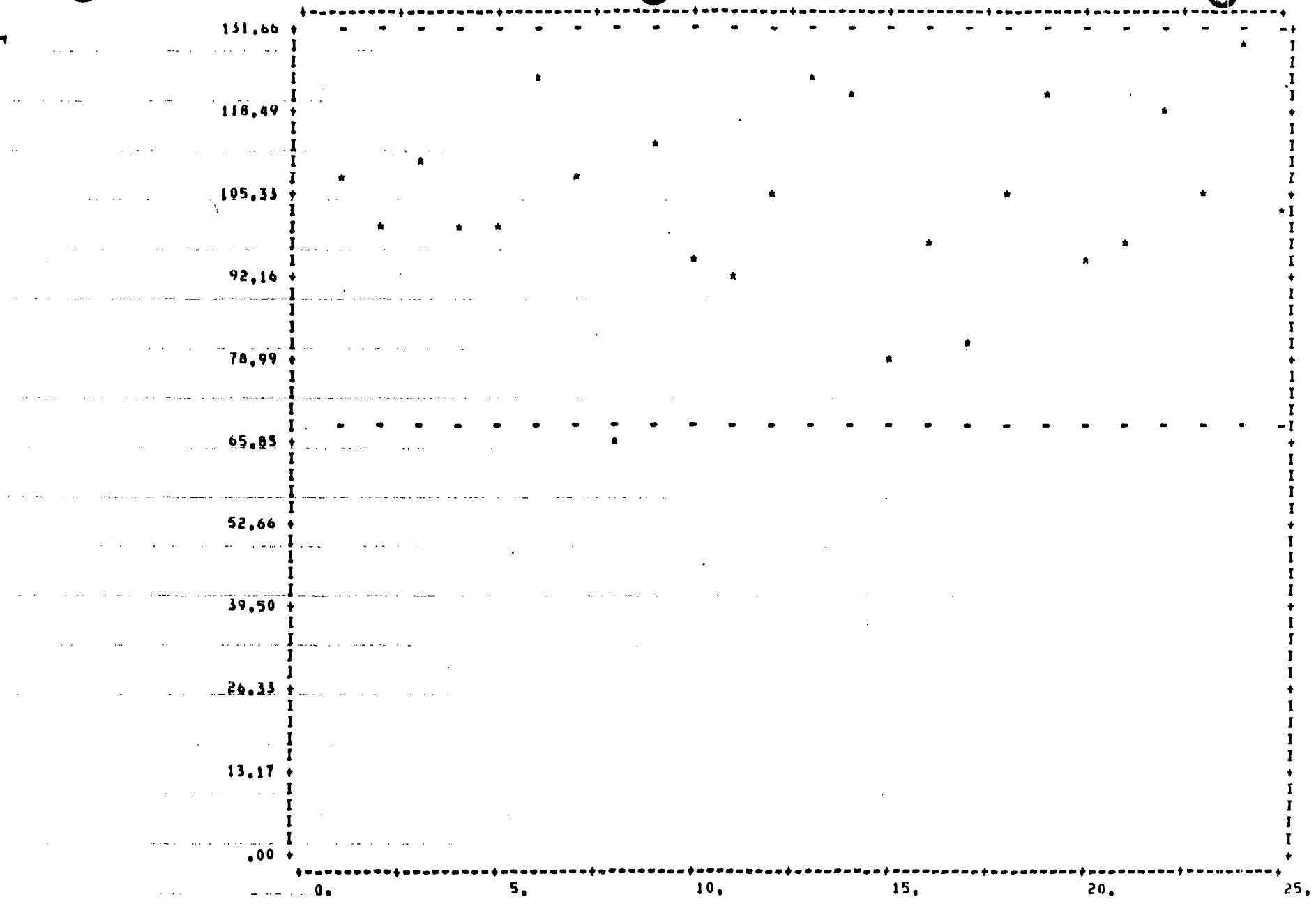
DCLEN12 SAMPLES 23250 THROUGH 50867

(*) - QC VALUES (-) -WARNING LIMITS

MEAN = 86.6590 STANDARD DEVIATION = 8.52285

MEDIAN = 87.0000 SKEWNESS = -.120015 KURTOSIS = 3.67436

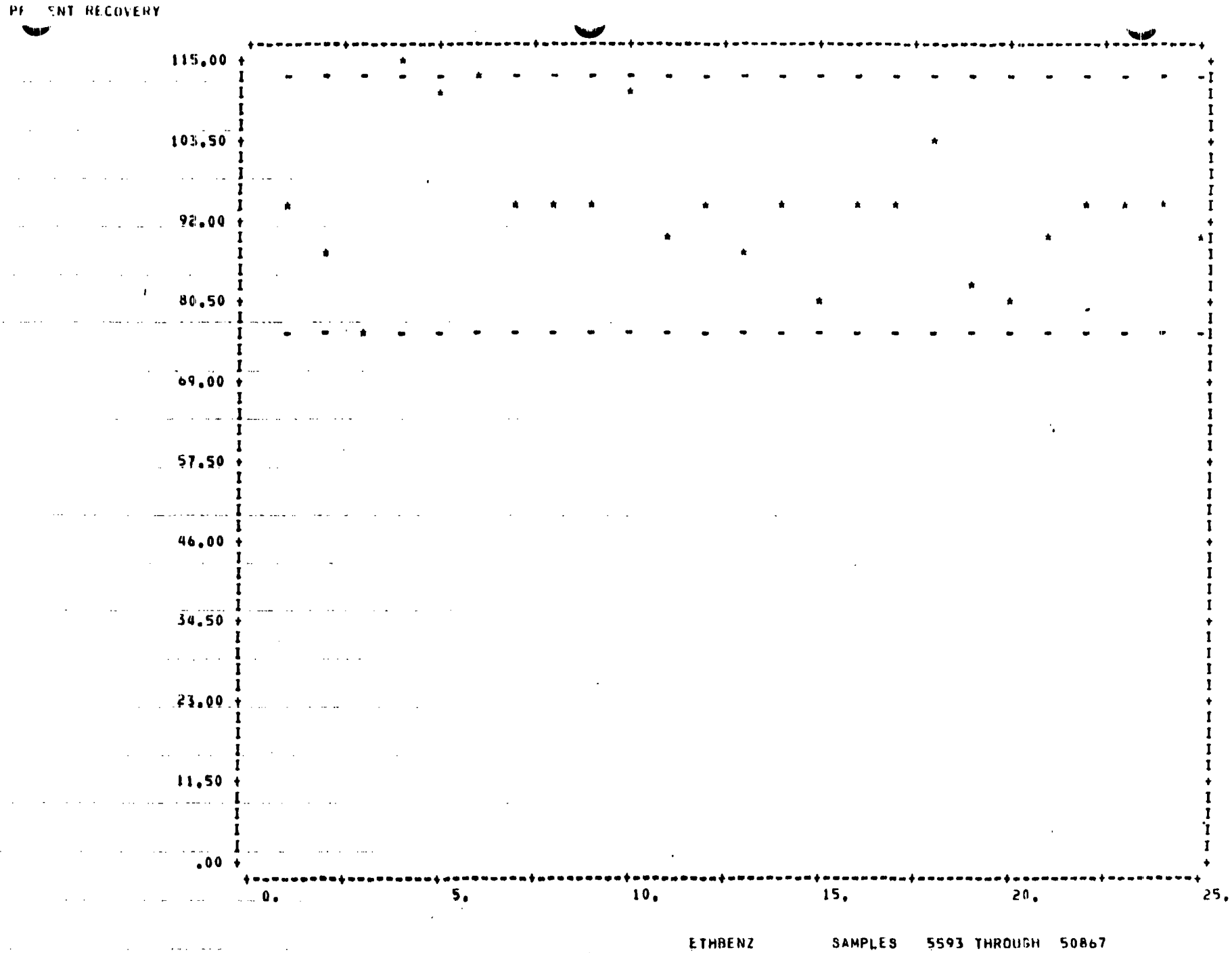
PERCENT RECOVERY



HG SAMPLES 64261 THROUGH 50904

(*) - QC VALUES (-) -WARNING LIMITS

MEAN = 99.1502 STANDARD DEVIATION = 15.7322
MEDIAN = 98.5798 SKEWNESS = .108757 KURTOSIS = 2.46181

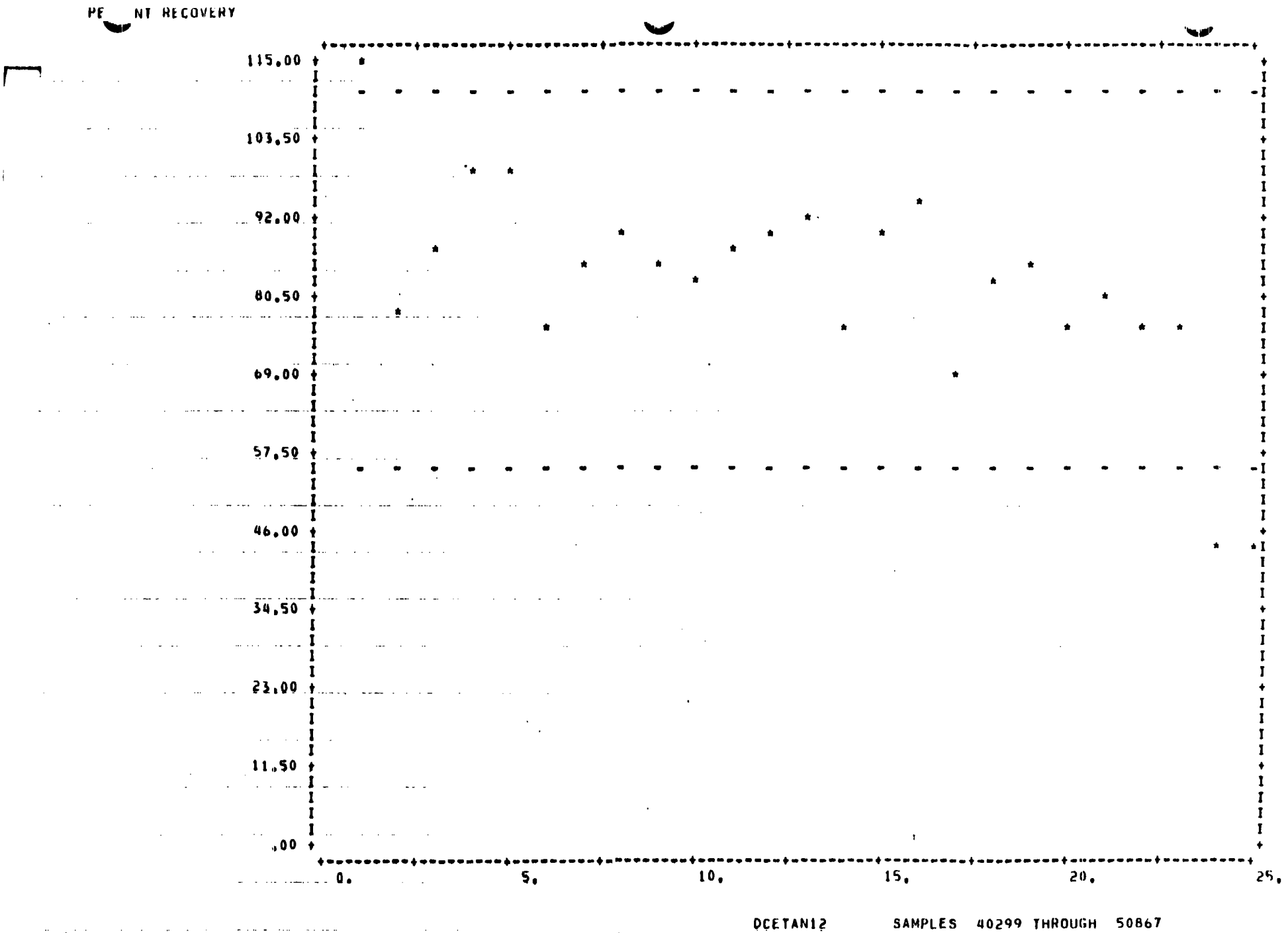


ETHBENZ SAMPLES 5593 THROUGH 50867

(*) - QC VALUES (-) -WARNING LIMITS

MEAN = 95.0119 STANDARD DEVIATION = 8.99539

MEDIAN = 95.0000 SKEWNESS = .196932E-02 KURTOSIS = 3.07061



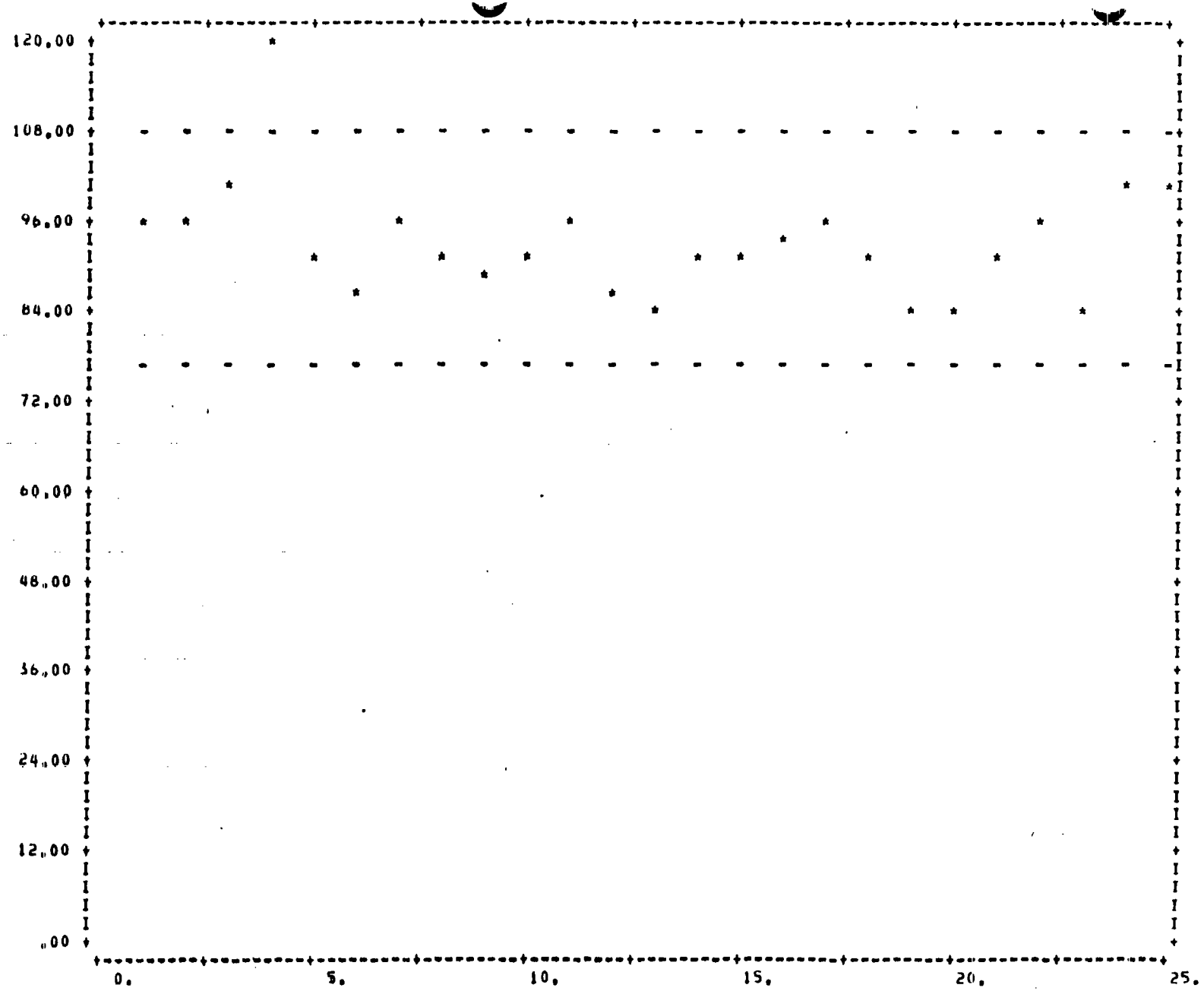
DCETAN12 SAMPLES 40299 THROUGH 50867

(*) - QC VALUES (-) -WARNING LIMITS

MEAN = 82.4609 STANDARD DEVIATION = 14.1964

MEDIAN = 82.5000 SKEWNESS = -.829153E-02 KURTOSIS = 4.72746

PE' NT RECOVERY

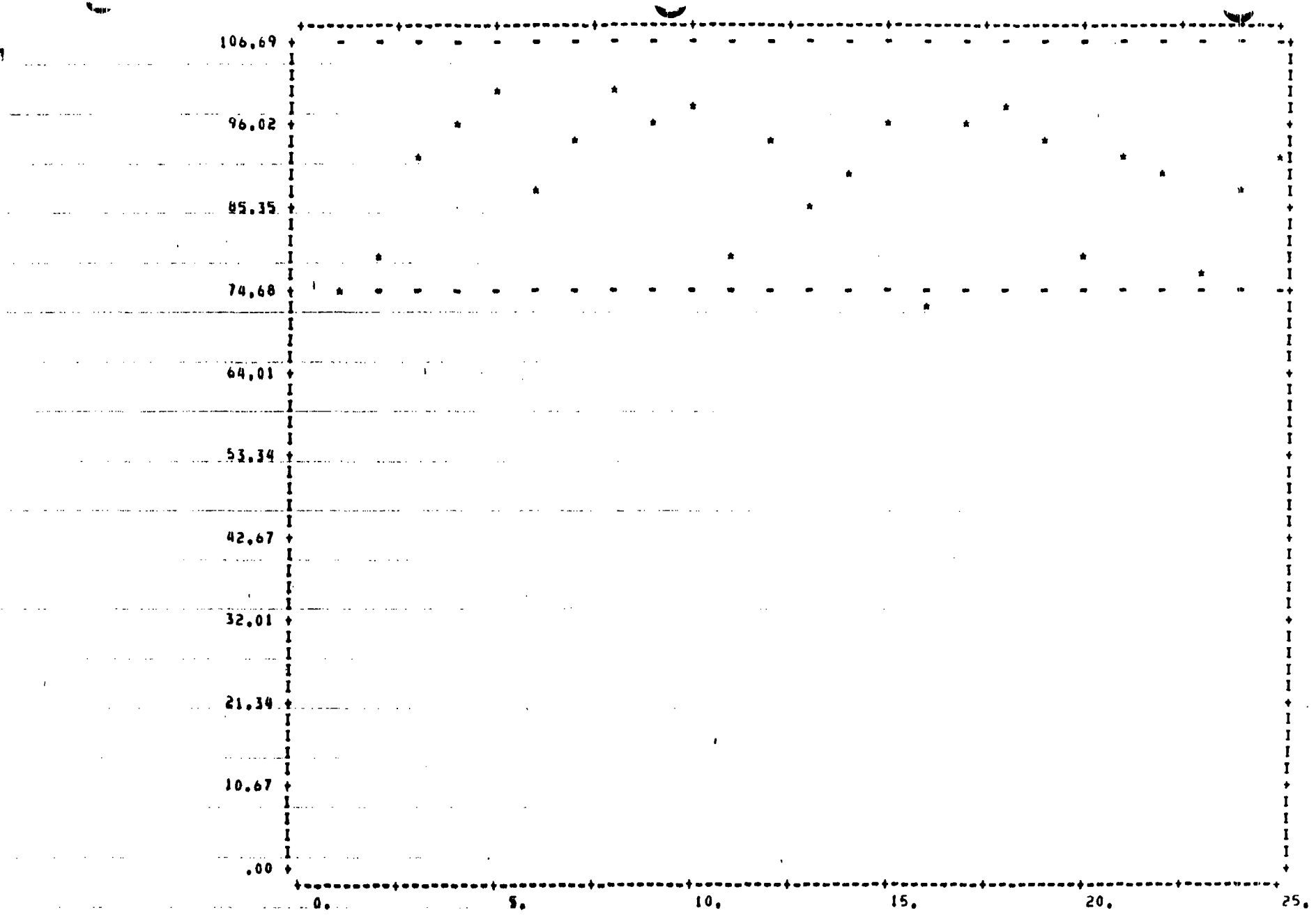


CLOROBZ SAMPLES 44382 THROUGH 50867

(*) - QC VALUES (-) -WARNING LIMITS

MEAN = 92.7198 STANDARD DEVIATION = 7.43594
MEDIAN = 91.0000 SKEWNESS = .693866 KURTOSIS = 8.25299

PERC[illegible] RECOVERY



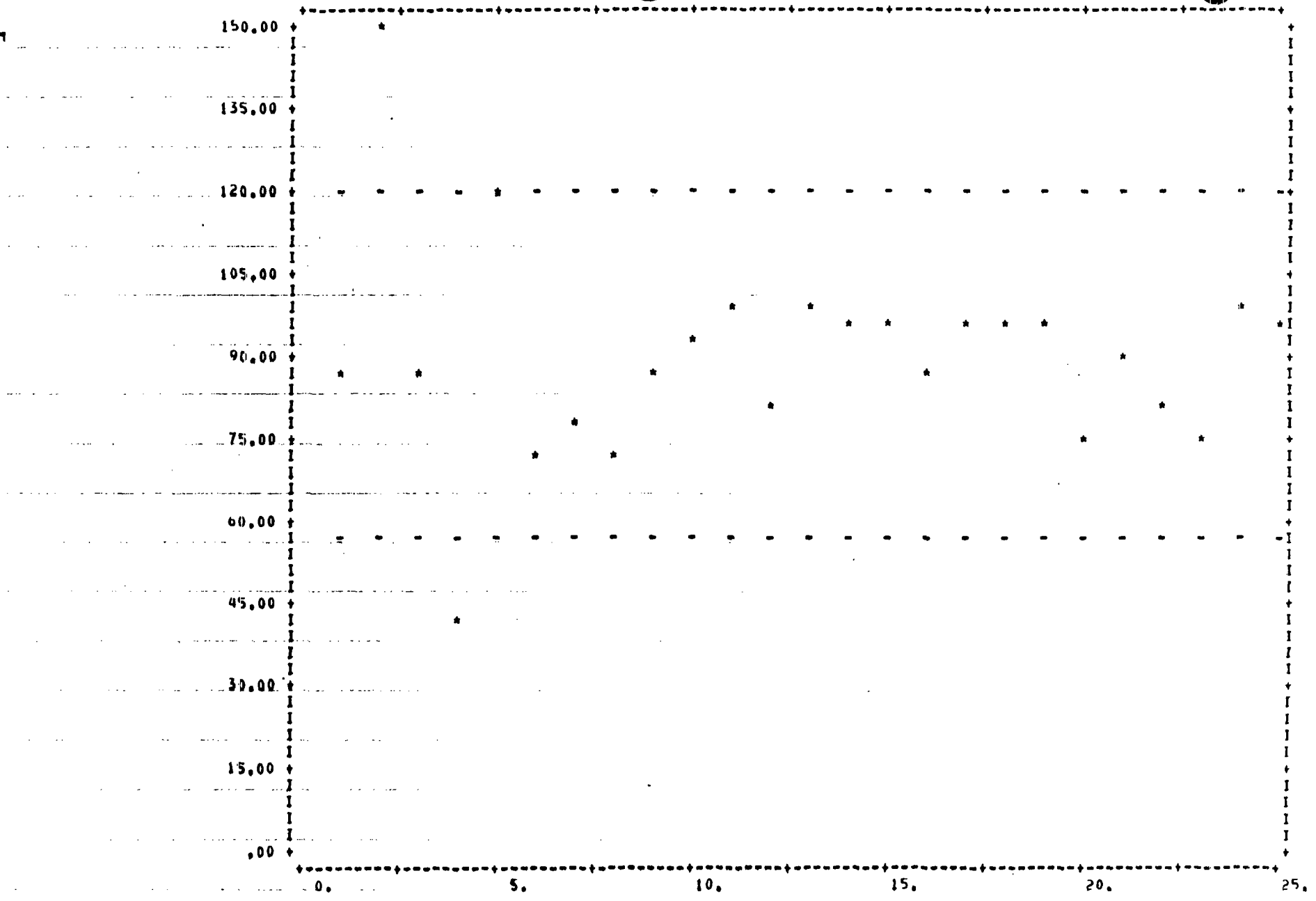
CH2CL2 SAMPLES 23250 THROUGH 50867

(*) - QC VALUES (-) -WARNING LIMITS

MEAN = 90.7871 STANDARD DEVIATION = 7.94928

MEDIAN = 92.5000 SKEWNESS = -.646415 KURTOSIS = 2.47973

P ENT RECOVERY

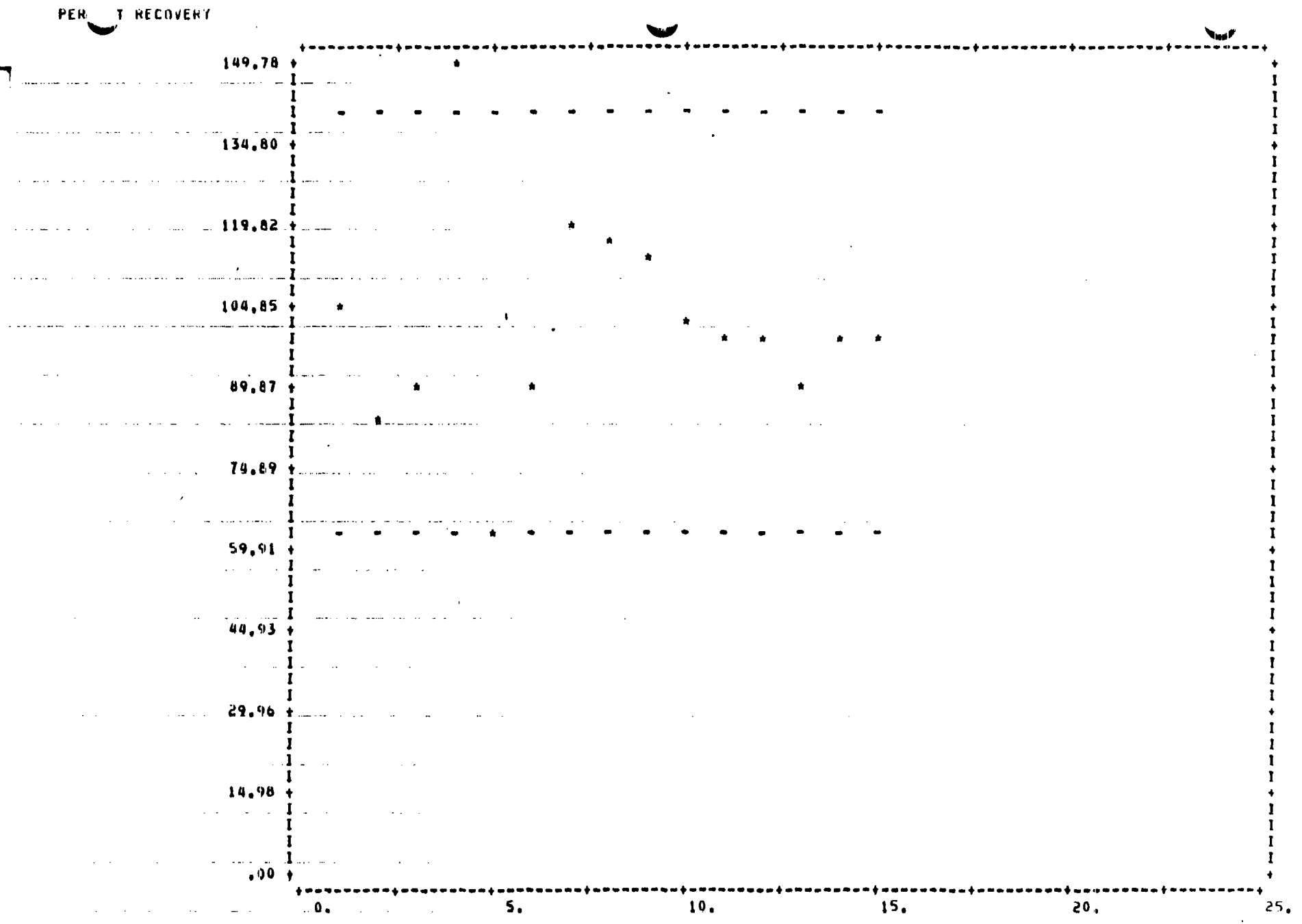


CL3CCH3 SAMPLES 39705 THROUGH 50867

(*) = QC VALUES (-) =WARNING LIMITS

MEAN = 88.5001 STANDARD DEVIATION = 15.9869

MEDIAN = 87.5000 SKEWNESS = .187665 KURTOSIS = 8.46347

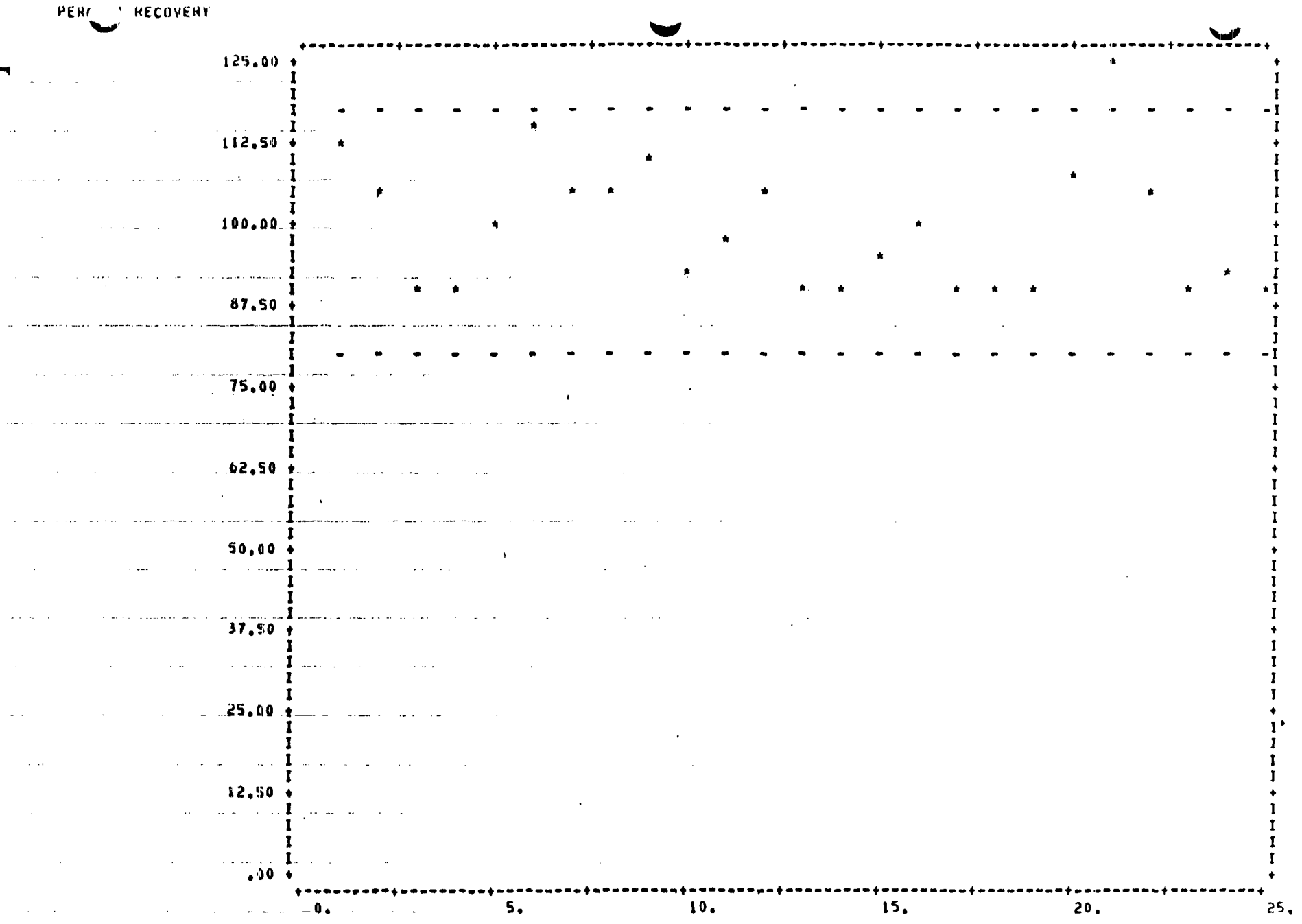


CL4C2 SAMPLES 39705 THROUGH 50867

(*) - QC VALUES (-) -WARNING LIMITS

MEAN = 101.354 STANDARD DEVIATION = 19.6983

MEDIAN = 100.000 SKEWNESS = .206263 KURTOSIS = 4.25532



TOLUENE SAMPLES 29830 THROUGH 50867

(*) - QC VALUES (-) -WARNING LIMITS

MEAN = 98.1067 STANDARD DEVIATION = 9.36984

MEDIAN = 97.0350 SKEWNESS = .343119 KURTOSIS = 3.13778

FIGURE 6

STATISTICS PROGRAM

```
STATISTICS FOR HG          USING ALL 54 SAMPLES
MEAN = 99.1502  STANDARD DEVIATION = 15.7322
MEDIAN = 98.5798  SKEWNESS = .108757  KURTOSIS = 2.46181
WARNING LIMITS ARE 67.6858  AND 130.615
CONTROL LIMITS ARE 51.9536  AND 146.347
```

```
STATISTICS FOR PB          USING ALL 190 SAMPLES
MEAN = 106.140  STANDARD DEVIATION = 11.6732
MEDIAN = 105.064  SKEWNESS = .276566  KURTOSIS = 5.64324
WARNING LIMITS ARE 82.7938  AND 129.487
CONTROL LIMITS ARE 71.1205  AND 141.160
```

```
STATISTICS FOR ZN          USING ALL 157 SAMPLES
MEAN = 106.180  STANDARD DEVIATION = 11.8682
MEDIAN = 105.128  SKEWNESS = .265860  KURTOSIS = 4.82146
WARNING LIMITS ARE 82.4436  AND 129.916
CONTROL LIMITS ARE 70.5755  AND 141.784
```

```
STATISTICS FOR BENZ        USING ALL 45 SAMPLES
MEAN = 98.2431  STANDARD DEVIATION = 8.50822
MEDIAN = 100.000  SKEWNESS = -.619471  KURTOSIS = 3.10783
WARNING LIMITS ARE 81.2267  AND 115.260
CONTROL LIMITS ARE 72.7185  AND 123.768
```

```
STATISTICS FOR BENZ        USING ALL 145 SAMPLES
MEAN = 97.5209  STANDARD DEVIATION = 11.8702
MEDIAN = 99.0000  SKEWNESS = -.373827  KURTOSIS = 4.19214
WARNING LIMITS ARE 73.7806  AND 121.261
CONTROL LIMITS ARE 61.9104  AND 133.131
```

FIGURE 7

SPIKED RECOVERY CONTROL CHART

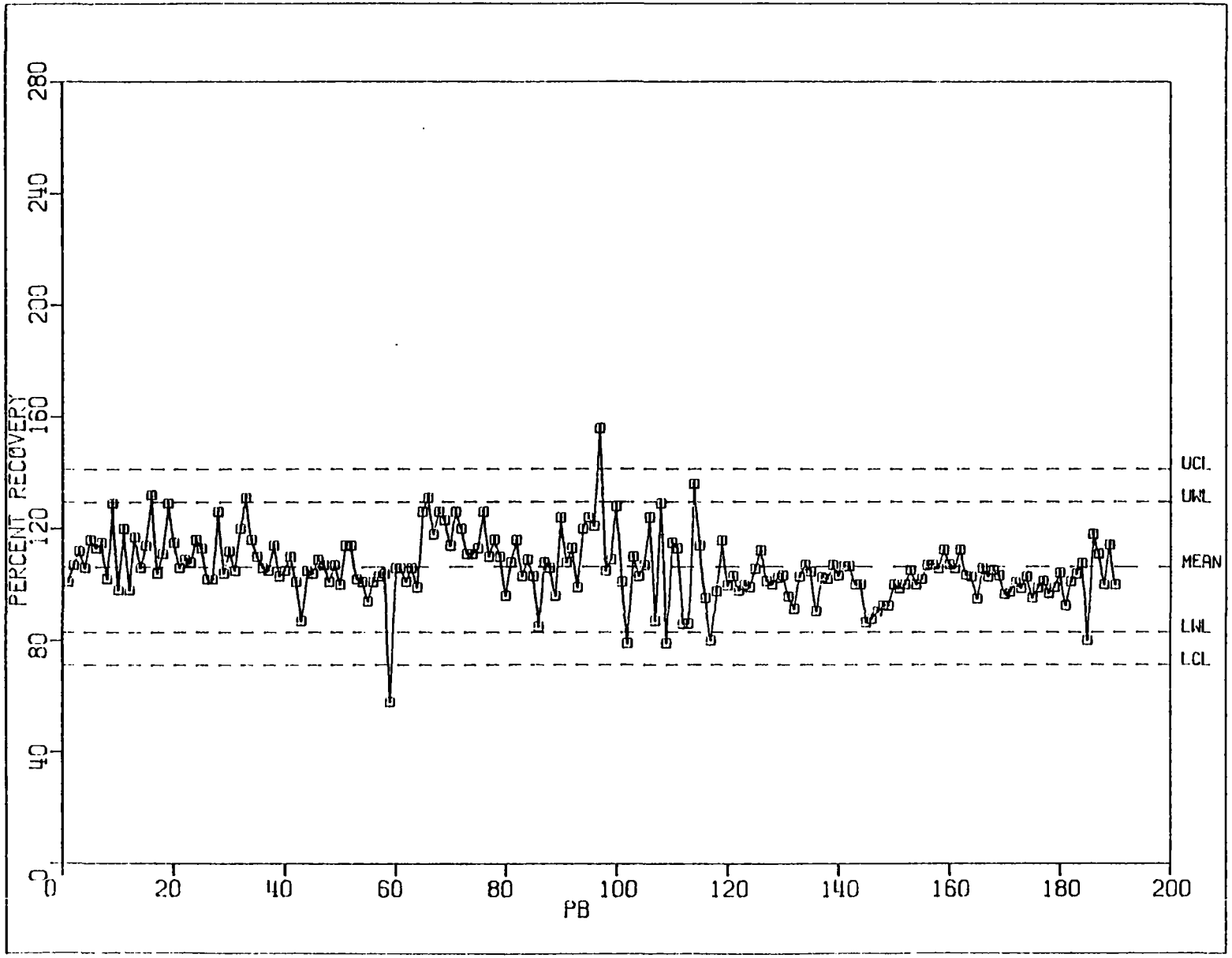


FIGURE 8

WARNING PROGRAM

```
*** QC WARNING PROGRAM ***

Enter parameter name ?BENZ
Enter QC type (3,50,51,etc.) ?51

STATISTICS FOR BENZ     USING ALL 145 SAMPLES
MEAN = 97.5209  STANDARD DEVIATION = 11.8702
WARNING LIMITS ARE 73.7806  AND 121.261
CONTROL LIMITS ARE 61.9104  AND 133.131

  SAMPLE      VALUE
-----------------------
   97273   128.000
   97673   130.000
   98150   63.0000
   98670   63.0000
   99578   73.0000
   28036   63.0000
   28684   65.0000
   12298   123.077
   98104   70.0000
   99584   68.0000
   17709   122.581

Enter parameter name ?ZN
Enter QC type (3,50,51,etc.) ?50

STATISTICS FOR ZN       USING ALL 157 SAMPLES
MEAN = 106.180  STANDARD DEVIATION = 11.8682
WARNING LIMITS ARE 82.4436  AND 129.916
CONTROL LIMITS ARE 70.5755  AND 141.784

  SAMPLE      VALUE
-----------------------
   67731   75.0000
   90477   133.000
   91039   133.000
   91132   139.000
   91952   76.0000
   91959   77.0000
   93181   133.000
   95987   140.000
   76738   132.000
   98496   135.000
   98950   67.0000
   26161   66.6667
   86390   101.147

STATISTICS FOR BENZ     USING ALL 45 SAMPLES
MEAN = 98.2431  STANDARD DEVIATION = 8.50822
WARNING LIMITS ARE 81.2267  AND 115.260
CONTROL LIMITS ARE 72.7185  AND 123.768

  SAMPLE      VALUE
-----------------------
    5718   120.000
   17857   77.4193
   18234   80.6451

Enter parameter name ?PB
Enter QC type (3,50,51,etc.) ?50

STATISTICS FOR PB       USING ALL 190 SAMPLES
MEAN = 105.140  STANDARD DEVIATION = 11.6732
WARNING LIMITS ARE 82.7938  AND 129.487
CONTROL LIMITS ARE 71.1205  AND 141.160

  SAMPLE      VALUE
-----------------------
   84864   132.000
   62095   131.000
   89182   56.0000
   94674   131.000
   98215   156.000
   98591   79.0000
    2412   79.0000
    2409   136.000
   26277   80.0000
   17569   80.0000

Enter parameter name ?HG
Enter QC type (3,50,51,etc.) ?50

STATISTICS FOR HG       USING ALL 52 SAMPLES
MEAN = 99.1502  STANDARD DEVIATION = 15.7322
WARNING LIMITS ARE 67.6850  AND 130.615
CONTROL LIMITS ARE 51.9528  AND 146.347

  SAMPLE      VALUE
-----------------------
   17683   67.0000
   22504   131.657

Enter parameter name ?
```

ATTACHMENT 5

ANALYTICAL PROCEDURES FOR CHLORINATED DIBENZO-P-DIOXINS AND DIBENZOFURANS

ACKNOWLEDGMENT

The author thanks J. C. T. Hollander for helpful discussions.

Registry No. H_2SO_4, 7664-93-9.

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Determination of Part-per-Trillion Levels of Polychlorinated Dibenzofurans and Dioxins in Environmental Samples

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The analytical method permits determinations of parts-per-trillion levels and lower of tetrachloro through octachloro congeners of dibenzo-*p*-dioxins and dibenzofurans in various types of biological tissues and sediments. Preliminary tests also indicated the method is applicable to determinations of tetrachloro through hexachloro congeners of ortho-unsubstituted polychlorinated biphenyls. Interferences both from biogenic and from xenobiotic substances are reduced to extremely low levels. The procedure has an extremely low susceptibility to false-positive determinations which could result from the presence of a wide variety of cocontaminants. A modular approach to contaminant enrichment has permitted the integration of seven processes into a two-step procedure, significantly reducing time requirements and the number of sample manipulations, and making the procedure amenable to automation. The reliability and accuracy of the procedure are demonstrated by the results of intralaboratory and interlaboratory studies and by successful analyses of over 200 samples of a wide variety of types.

Polychlorinated dibenzo-*p*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), and ortho-unsubstituted polychlorinated biphenyls (non-ortho PCBs) are three structurally and toxicologically related families of anthropogenic chemical compounds that have in recent years been shown to have the potential to cause serious environmental contamination (*1–6*). These substances are trace-level components or byproducts in several large-volume and widely used synthetic chemicals, principally PCBs and chlorinated phenols (*7, 8*), and can also be produced during combustion processes (*3, 9–11*) and by photolysis (*12, 13*). In general, PCDDs, PCDFs, and non-ortho PCBs are classified as highly toxic substances (*14*), although the toxicities are dramatically dependent on the number and positions of the chlorine substituents (*15*). About 10 individual members of a total of 216 PCDDs, PCDFs, and non-ortho PCBs are among the most toxic man-made or natural substances to a variety of animal species (*1–4*). The toxic hazards posed by these chemicals are exacerbated by their propensity to persist in the environment (*16–18*) and to readily bioaccumulate (*19–21*), and although the rate of metabolism and elimination is strongly species dependent (*20*), certain highly toxic isomers have been observed to persist in the human body for more than 10 years (*22*).

The majority of scientific and governmental concerns for the hazards of these compounds have been directed toward analytical methodologies, toxicology, epidemiology, and determination of the disposition in the environment of the single most toxic isomer, 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD) (*1–6, 8*).

More recently, however, investigations into the formation and occurrence of PCDFs suggest that this family of toxic compounds may commonly occur at comparable or greater levels than and could generally pose a greater hazard than PCDDs. PCDFs are often found as cocontaminants in and are readily produced from pyrolysis of PCBs (*7, 23–26*). Most important, the PCDFs produced from pyrolysis of PCBs are predominantly the most toxic isomers, those having a 2,3,7,8-chlorine substitution pattern (*5*). A number of recent fires involving electrical transformers and capacitors have demonstrated the potential for formation of hazardous levels of PCDFs from pyrolysis of PCBs (*26–28, 30*).

In light of these findings and because of the dearth of data pertaining to the occurrence of these compounds in the environment, PCDFs and non-ortho PCBs were included as target compounds in a proposed survey by this laboratory of important U.S. rivers and lakes for PCDDs. The decision to include as many PCDD isomers as possible was based on

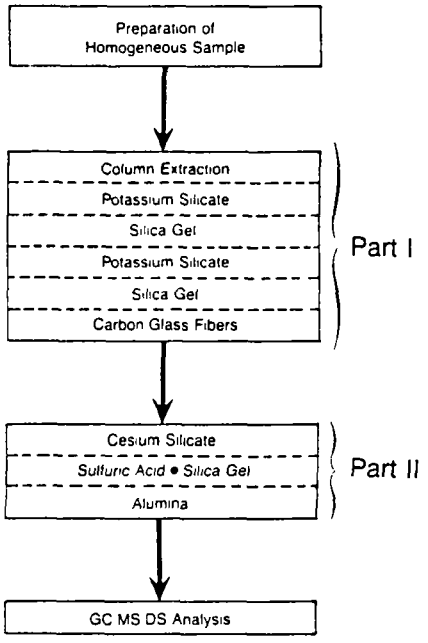

Figure 1. Flow chart of total procedure.

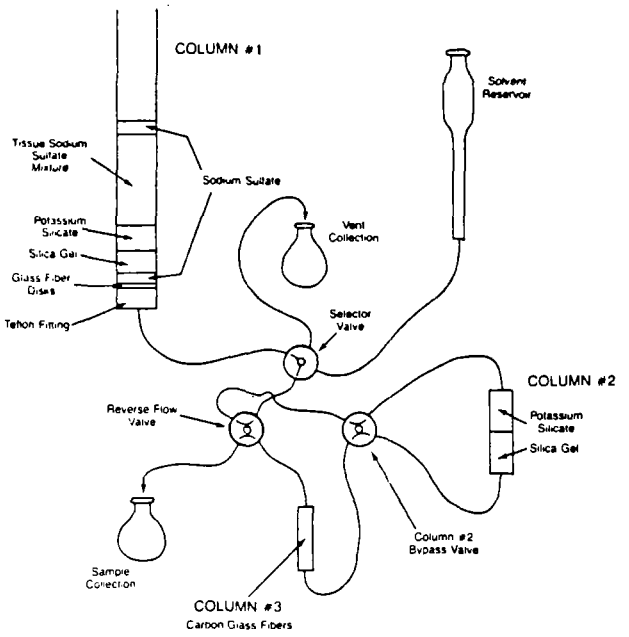

Figure 2. Schematic of part I enrichment apparatus.

several facts: (1) several other PCDD isomers are also extremely toxic (*15*); (2) pentachlorophenol, a large-volume fungicide and wood preservative, contains relatively high levels of hexa-, hepta-, and octachlorodibenzodioxins and essentially no TCDDs (*7, 8, 29*); and (3) incineration of materials containing chlorophenols readily produces mixtures of PCDDs, but 2,3,7,8-TCDD is a minor component. On the other hand, the highly toxic 1,2,3,7,8-pentachloro isomer is a major component of PCDD incineration products of pentachlorophenol (*7, 30*). Component-specific analyses can be a crucial link to the source of contamination because different sources of PCDDs and PCDFs usually produce mixtures of distinctly different relative component abundances (*7*). On the other hand, the preferential accumulation of certain isomers in animals may prevent source identification from analyses of biological samples.

The analytical method developed for this investigation was required to meet six criteria: (1) permit determinations of the majority of these compounds, especially those possessing more than three chlorine substituents; (2) permit isomer-specific determinations of the most toxic or otherwise important components; (3) provide a lower limit of detection for individual components of between 1 and 5 parts per trillion (pptr) in a variety of environmental samples; (4) generate data with an acceptable and adequately defined level of accuracy and precision; (5) exhibit a very low and well-defined susceptibility to interference and false-positive determinations; and (6) minimize analyst's time requirements to permit analyses of large numbers of samples.

Determinations of PCDDs and PCDFs demand an unusually high level of analytical assurance, not only because of the potential hazards of these compounds, the intensity of public and industry concern, and the widespread nature of the problem, but also because of the great difficulties in rigorous identification of individual isomers. These difficulties are not so much related to the problems of distinguishing between isomers—this problem is essentially solved (*31–34*)—but are related to the possibility of specific and nonspecific interferences from natural and especially xenobiotic substances (*35*).

Presented herein are the description of an analytical method and the results of validation and applications studies which define the accuracy and reliability and demonstrate the utility of the method developed for the determination of PCDDs, PCDFs, and non-ortho PCBs in a variety of environmental matrices.

EXPERIMENTAL SECTION

Enrichment Procedure. Tissue and sediment or soil samples (spiked with isotopic marker compounds) are processed in a two-part enrichment procedure (Figure 1). In part I, a mixture of the sample and sodium sulfate is subjected to solvent extraction, and the extract is, in the same process, passed through a series of silica-based adsorbents and then through the carbon/glass fiber adsorbent. The extract passes through the adsorbents in the following order: potassium silicate, silica gel, cesium or potassium silicate, silica gel, and finally an activated-carbon adsorbent. The residues of interest [PCDDs, PCDFs, and non-ortho PCBs, as well as other chemical classes such as polychlorinated naphthalenes (PCNs), polychlorinated biphenylenes, and certain polynuclear aromatic hydrocarbons] are retained on the carbon adsorbent and subsequently recovered by reverse elution with toluene.

In part II of the procedure, following a change of solvent to hexane, the sample is applied to a second series of adsorbents contained in two tandem columns. The first column contains small amounts of cesium or potassium silicate and sulfuric acid impregnated silica gel. The effluent from this column flows directly onto the second column containing activated alumina on which the final fractionation of several classes of residues is accomplished. Following reduction of sample volume, analyses are carried out by high-resolution gas chromatography/low-resolution mass spectrometry/computer data system analysis (HRGC/LRMS/DS) and under some circumstances by gas chromatography/electron capture detector analysis (GC/EC).

Part I. The components of the apparatus used in part I of the enrichment procedure are depicted in Figure 2. Column 1 (about 4.5 cm i.d. and about 1 m long) is connected to column 2 (22 mm i.d. × 100 mm, Michel–Miller precolumn 5769–34, Ace Glass, Vineland, NJ) and to column 3 (1.0 cm i.d. × 6 cm thick-walled, precision-bore glass tubing, Kontes, Vineland, NJ) by means of standard $^1/_{16}$ or $^1/_8$ in. o.d. Teflon tubing and tube end fittings. Column 3 is equipped with in-house fabricated Teflon fittings. The solvent flow switching valves are Hamilton miniature inert valves (Hamilton Co., Reno, NV): selector valve (no. 86781), on–off valve (no. 86775), and bypass and reverse-flow valves (no. 86781). The washing solvent reservoir is constructed of a 20-cm length of 12 mm o.d. glass tubing and a 200-mL reservoir fitted with a 24/40 female ground glass joint. The valving arrangement (Figure 2) is designed to enable the analyst to perform the following operations: venting of the solvent line from column 1, venting of the solvent reservoir, bypass of column 2, delivery of

the effluent from column 1 to columns 2 and 3 sequentially, delivery of solvent from the reservoir sequentially to columns 2 and 3 or to column 3 only, reversal of solvent flow in columns 2 and 3, and stoppage of solvent flow in all lines. The solvent reservoir is routinely pressurized with 1–10 psi nitrogen during column washings. Column 2 is packed with equal volumes, 15 mL each, of cesium or potassium silicate and silica gel (EM-60, 130 °C activated) bracketed by plugs of glass wool or preferably disks of glass fiber filters (3-μm retention GF/D, Whatman Inc., Clifton, NJ). Column 3 is packed with a mixture of Amoco PX-21 activated carbon and glass fibers as described previously (*36*). Column 1 is packed in the following sequence: two disks of glass microfiber filters (GF/D, 4.7-cm diameter, Whatman Inc., Clifton, NJ), a 2-cm depth of anhydrous sodium sulfate, 30 g of silica gel (EM-60, 130 °C activated), 30 g of potassium silicate (130 °C activated), 250 g of a 1:4 (w/w) mixture of the sample and anhydrous sodium sulfate, and lastly a 2-cm depth of anhydrous sodium sulfate.

Column 2 (Figure 2) is usually packed with fresh adsorbents for each sample, but this column can be used for more than one sample if the amounts of extracted materials, such as lipids, are small. The carbon adsorbent in column 3 is routinely reused following washings (under 3–8 psi of nitrogen) between samples with the following solvent sequence: 100 mL each in reverse flow of toluene, methanol, toluene, and cyclohexane/methylene chloride 50, 50 (solvent A). Column 2 is bypassed during these washings except for the final washing with solvent A, which is directed through column 2 in the reverse direction to remove residual air and possible contaminants. Care must be taken to avoid passing methanol through column 2. Another 100 mL of solvent A is routinely passed through columns 2 and 3 in the forward direction as the final solvent washing. Complete displacement of toluene from column 3 is essential. After columns 2 and 3 are properly washed and column 1 is loaded with adsorbents and sample, a solution (usually 100 μL) of marker compounds is applied to column 1 and washed onto the packings with four or five 20-mL (approximate) portions of solvent A using a Teflon wash bottle. The selector valve is positioned so that column 1 is connected to the vent line and air is allowed to escape. The flow of air from the column is monitored as it bubbles through solvent at the vent line exit. After the sample is spiked with marker compounds, 650 mL of solvent A is carefully applied to column 1, and the movement of the solvent front is observed. As the solvent front enters the transfer line (about 1 m in length), air bubbles in the line are removed by stopping the flow and tapping the line. When the solvent front reaches the selector valve, the valve is repositioned to direct the extract through columns 2 and 3, and the enrichment procedure is under way. The effluent is collected in a 1-L flask positioned above columns 2 and 3 to maintain a positive back-pressure on these columns. The height of column 1 above the collection flask is adjusted to produce a solvent flow of not more than 5 mL/min but sufficient to complete the process overnight. Occasionally the solvent flow will slow or stop during the initial stage and will require the application of 1 or 2 psi of gas pressure to the system at column 1. Rarely, the glass fiber filters on the inlet end of column 3 become clogged during the processing of decomposed or very oily (especially lake trout) samples. To reduce these complications, a removable column (1.0 cm i.d. × 2 or 3 cm) containing 4 or 5 disks of glass microfibers can be placed in line at the exit end of column 2. If this filter column becomes clogged, it can be replaced during the process.

Following completion of the initial extraction/adsorption operation, column 3 (bypassing column 2) is washed in the forward direction with 75 mL of solvent A and then 50 mL of methylene chloride/methanol/benzene (75/20/5) at a flow of approximately 5 mL/min. These washings are collected in the flask with the initial eluate. The reservoir is then charged with 40 mL of toluene which is passed through the carbon (column 3) in the reverse-flow direction at approximately 2 mL/min and collected in a 100-mL round bottom (24/40) flask. At this point, part I of the procedure is complete.

The sample in toluene is subjected to rotary evaporation at 55 °C under a vacuum of about 0.1–0.2 atm. The rotary evaporation system must be maintained in an uncontaminated condition by periodic washings with organic solvents. No lubricating greases can be used. The integrity of the sample is protected during rotary evaporation by the use of a vapor trap situated between the sample flask and the evaporation apparatus; the vapor trap is thoroughly washed with toluene between samples. The toluene solution (sample) is carefully reduced to less than 1 mL or just to dryness and removed immediately. The solution or dry sample can be stored in a freezer. At this point, the sample is ready for part II of the procedure (after removal of all toluene).

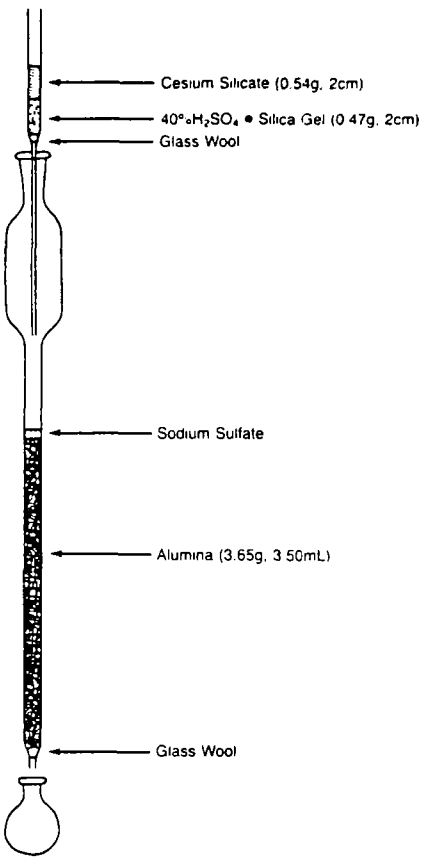

Figure 3. Schematic of part II enrichment procedure.

Part II. The apparatus for part II of the enrichment procedure consists of two columns arranged in tandem (Figure 3). Column 4 is prepared from a disposable Pasteur pipet and is packed first with a plug of glass wool, then with 3 cm (0.50 g) of sulfuric acid impregnated silica gel, then with 3 cm (0.54 g) of cesium potassium silicate (not heat activated), and finally with 0.5 cm of anhydrous sodium sulfate. Column 5 is constructed from a 5-mL graduated pipet fitted with a 20-mL reservoir and a ground-glass joint. Column 5 is packed with a plug of glass wool followed by 3.50 mL (3.65 g) activated (190 °C) alumina and then 0.5 cm of anhydrous sodium sulfate. The alumina is packed firmly by sharply tapping the supporting clamp.

Columns 4 and 5 (Figure 3) are thoroughly washed before use, column 4 with 10 mL of hexane and column 5 under approximately 5 psi of nitrogen pressure, with 30–50 mL of hexane to remove entrapped air. Following the washings, column 4 is partly inserted into column 5 so that the effluent from column 4 flows directly onto the adsorbent bed of column 5. A 50-mL collection vessel is placed at the exit end of column 5. Pasteur pipets previously heated for several hours at 500 °C are used for liquid transfers. The sample is applied to column 4 by using four to six separate 1-mL washings (approximate volumes) of hexane totaling 5.0 mL. Each washing is allowed to pass through column 4 and completely onto the alumina of column 5 before the next washing is applied. After 5.0 mL of hexane has passed through column 4, this column is discarded, and a second 5.0-mL volume of hexane is then applied to column 5. The following sequence of eluting solvents is then applied to column 5: 15 mL of 2%, then 15 mL of 5%, and finally 20 mL of 8% methylene chloride in hexane. A total of 60 mL of effluent is thus collected in two fractions, the first measuring 23 mL and the second 37 mL. Due to variations in the activities of different lots of alumina, the

correct elution volumes must be carefully determined for each lot.

The second fraction, containing the residues of interest, is reduced in volume to about 0.5 mL under a stream of nitrogen in a 40 to 45 °C water bath. The sample is transferred to a conical minivial with four 0.5-mL washings with methylene chloride, each washing being reduced to a smaller volume under a stream of nitrogen before the next is added. Following the last transfer, the solvent is completely evaporated and the appropriate volume of desired solvent (usually 10 μL of toluene or o-xylene) is added just prior to analysis. If the analysis is to be performed later, the sample can be kept in the dry state and stored in a freezer. Before the sample is injected, the solution is drawn up into the microliter syringe and applied repeatedly to the wall of conical portion of the vial to bring the entire sample into solution. Gas chromatographic/mass spectrometric analyses are carried out by the direct injection technique (no splitting of the sample) with 2–4-μL injection volumes or by the on-column technique in which 1–2-μL volumes are injected.

Sample Preparation. Tissue and sediment samples are combined with at least 4 times their weight of anhydrous sulfate. Tissue samples are first cut into small pieces, ground in a meat grinder (if necessary), and mixed thoroughly with anhydrous sodium sulfate with a spoon in a glass or polyethylene dish. The mixture is then spread out to a depth of less than 3 cm so that the mass, which solidifies after 3–6 h, can be readily broken up after drying overnight. The mixture is then dry-blended (any kitchen model blender) in a glass jar to yield a fine powder. Samples of low water content did not require overnight equilibration with sodium sulfate before blending. A second blending of the mixture 4–6 h after the first is often required to produce a more homogeneous and finely divided sample.

Analytical Instruments and Conditions. Determinations of PCDFs and PCDDs were carried out with a Finnigan 4023 GC/MS system equipped with an INCOS data system and with negative and positive chemical ionization options. Methane was used as the reagent gas for the negative ion chemical ionization analyses. The gas chromatograph was usually fitted with either a 30 m × 0.25 mm DB-5 fused-silica capillary column (J&W Scientific, Inc., Rancho Cordova, CA) or a 55 m × 0.27 mm Silar 10 c column prepared by H. R. Buser, Swiss Federal Research Station, Wadenswil, Switzerland. The carrier gas was helium and the following temperature program was routinely used with o-xylene solvent: 150–255 °C at 3 °C/min and then 12 °C/min to 290 °C and hold for 10 min. The electron impact mode (EI) and multiple ion detection (MID) were routinely used for GC/MS identification and quantitation of PCDFs and PCDDs including isotopic marker compounds ([^{13}C]-TCDD, [^{37}Cl]-TCDF, and [^{37}Cl]-OCDD). By use of DB-5 column, a series of either 8 or 12 mass-to-charge ratio (m/z) values were monitored within each of five or six chromatographic windows, each window being defined by the lower and upper elution limits of a particular group of PCDF and PCDD congeners. The MID analysis usually involved the monitoring of four or five members of a molecular ion cluster and occasionally of the fragment ion cluster resulting from the loss of COCl, M − 63.

Gas chromatographic analyses employing a packed column [2 m × 2 mm 3% OV-17 on 100/120 Supelcoport (Supelco, Inc., Bellefonte, PA)] were carried out on a Varian 3700 gas chromatograph equipped with an electron capture detector. Nitrogen was used as the carrier gas with the following temperature program: 180–270 °C at 8 °C/min and hold for 15 min.

Materials. All solvents were glass distilled grades (MC/B, Cincinnati, OH, or Burdick and Jackson, Muskegon, MI). Silica Gel 60, 70–230 mesh (EM Reagent, MC/B, Cincinnati, OH) and acid alumina (AG4, Bio Rad Labs, Richmond, CA) were used. Alumina was washed with methanol and then methylene chloride and oven activated at 190 °C for at least 2 days. Silica gel was washed in the same manner and activated at 130 °C. The silica gel was stored in the 130 °C oven and removed just prior to use. Sodium sulfate (MC/B, no. SX760) is heated at 500 °C overnight and stored in screw capped bottles.

Amoco PX-21 activated carbon was obtained from the Amoco Research Center, Naperville, IL 60566, and lot numbers 75-8, 75-11, 76-16, and 78-10 were successfully used in this laboratory. This carbon is now commercially available from Anderson Development Co., Adrian, MI 49221, under the name AX-21.

Potassium and cesium silicates were prepared from the reaction of the corresponding alkali metal hydroxides with silica gel in methanol at 55 °C for 90 min. The reaction is carried out in a 1- or 2-L round-bottom flask which is rotated and heated with a rotary evaporation apparatus (no vacuum applied). Sixty grams of CsOH (99+%, Aldrich Chemical Co., Milwaukee, WI) is dissolved in 200 mL of methanol and separated from insoluble material by decantation. An additional 200 mL of methanol is added followed by 100 g of silica gel. For potassium silicate, 168 g of KOH (J. T. Baker Chemical Co., Phillipsburg, NJ), 300 g of silica gel (EM60), and approximately 700 mL of methanol are used; decantation is not necessary for KOH. Following the reaction, the mixture is poured into a large glass column containing a plug of glass wool. Special care must be exercised to avoid contact with the extremely caustic solution, especially eye contact. The adsorbent is washed into the column with methanol, and then 200 mL of methanol for every 100 g of silica gel is added to the column. The methanol can be pushed through the column under slight gas pressure, and as the level of the liquid reaches the bed of adsorbent, 200 mL of methylene chloride for every 100 g of silica gel is applied. The liquid is pushed through the column and the silicate partly or completely dried under a slow flow of nitrogen. Cesium silicate is dried completely under a stream of nitrogen and is not heat activated; potassium silicate is activated at 130 °C.

Sulfuric acid impregnated silica gel (40% w/w), abbreviated as SA·SG, is prepared by adding 2 parts of concentrated sulfuric acid to 3 parts by weight of 130 °C activated silica gel in a screw capped bottle and shaking until the mixture is completely free of lumps, about 15 min. The silica gel is activated at 130 °C; unactivated silica gel is unsatisfactory for the preparation of SA·SG. The adsorbent is stored in a screw capped bottle.

Nitrogen gas used for evaporations of solvents is passed through a copper tube (40 mm o.d. × 60 cm) containing activated carbon (coconut charcoal, Fisher Scientific Co., Pittsburgh, PA) bracketed by glass wool and glass microfiber filters. Following the carbon trap, a microfiber filter (Microfibre filter 9802-AAQ, 505-AAQ, 0.3-μm retention, Balston Filter Products, Lexington, MA) is inserted in the line in an attempt to prevent movement of carbon particles through the nitrogen line.

RESULTS AND DISCUSSION

Development and Functions of the Components of the Enrichment Procedure. *Part I.* A primary objective in the initial approach to the development of this method was to make optimum use of the highly selective absorbtivity of activated carbons for polychlorinated polycyclic aromatic compounds (*37*). The carbon adsorbent selected for this procedure was Amoco PX-21 dispersed in glass fibers (CGF) which has been thoroughly evaluated in this laboratory with regard to its selectivity for a wide variety of chemical classes (*36*, *37*). At least four lots of PX-21 carbon have been successfully employed by this and other laboratories (*26*, *38–46*) in analyses of PCDDS and PCDFs.

Application of extracts of whole fish directly to the carbon absorbent dispersed in glass fibers was found to be generally unacceptable due to the adsorption of biogenic substances causing high back pressures. Pretreatment of the tissue extract with the strongly basic absorbent potassium silicate (KS) (*47*, *48*) followed by activated silica gel (SG) greatly facilitated the flow of the tissue extract through the carbon adsorbent. Other combinations with alumina and with Florisil or with potassium silicate alone were less effective. The combination of KS, SG, and PX-21 carbon adsorbents achieved a very high degree of enrichment of PCDDs, PCDFs, and non-ortho PCBs. Tissue samples up to 50 g and containing 10–20 g of fat routinely give only submilligram residues in the sample recovered by reverse elution of the carbon with toluene. Integration of these three steps yielded a procedure that permitted simultaneous sample extraction, removal of acidic and highly polar coextractables, and selective adsorption of the compounds of interest onto carbon (part I) and was readily

adapted to a modular arrangement which simplified sample, solvent, and adsorbent manipulations (Figure 2). Several sets of apparatus could each be loaded with a sample, the three adsorbents, and solvent, and the enrichment processes allowed to proceed unattended, by gravity solvent flow. The use of a second combination of potassium silicate and silica gel (column 2, Figure 2) ensures that the interfering lipid materials are prevented from reaching the carbon and permits the analyst to visually estimate the amount of colored lipid material which is being adsorbed by the potassium silicate/silica gel combination. In those cases in which little or no accumulation of colored material is observed on column 2, consideration can be given to reusing column 2 for another sample. Cesium silicate retains acidic compounds more effectively than KS and was initially used in column 2 but is 50 times more costly.

The combined operations of part I eliminate the need for procedures which require extensive sample manipulations and which are more labor intensive. Such procedures which are commonly employed in other methods include one or more of the following: (1) acidic or basic digestion of the sample, (2) multiple liquid–liquid partitioning steps, (3) Soxhlet extraction, or (4) gel permeation chromatography. The ability to effect multiple enrichment procedures in a one-step, continuous operation can result in enhanced recovery and precision, in addition to reduced analysis time. Furthermore, this operation lends itself to the possibility of development into an automated, multisample procedure (*49*).

Gel permeation chromatography (GPC) was initially employed in this procedure as an enrichment step preceding the carbon adsorbent but often did not have the capacity for the large samples required in these analyses. Furthermore, the incorporation of GPC into the initial enrichment procedure necessitated additional sample extraction and solvent volume reduction steps precede the GPC procedure.

In addition to protecting the adsorptive capacity of the activated carbon, the silicate adsorbent has been demonstrated in this laboratory to remove acidic compounds which represent potentially serious interferences to determinations of PCDDs and PCDFs. The silicate adsorbents retain substances which exhibit pK_A acidity constants of 10 and lower, including phenolic and carboxylic acid compounds and sulfonamides (*48*). In particular, hydroxy PCBs and hydroxydiphenyl ethers, compounds which can produce false-positive GC/MS responses, are effectively removed by the silicates (*35*).

Under the conditions of this enrichment procedure, the carbon adsorbent will retain only a limited number of classes of organic compounds (*50*), including polyhalogenated planar multi-ring aromatic compounds, to some extent PAHs with more than three rings, and strongly acidic compounds that are effectively sequestered by the silicate adsorbent before reaching the carbon. The large majority of synthetic organic compounds which are commonly encountered as persistent environmental contaminants are weakly adsorbed and readily displaced from the carbon by the extraction solvent. Included in this group of chemicals are compounds which interfere in GC/MS determinations of PCDDs, PCDFs, and non-ortho PCBs, such as DDE, PCBs, methoxy PCBs, polychlorinated diphenyl ethers (PCDPEs), and methoxy PCDPEs (*35*). The carbon adsorbent also exhibits a very low affinity for the biogenic substances which are not retained by the potassium silicate/silica gel combination.

Part II. In part II of the enrichment procedure (Figure 3) the sample is first passed through a strongly basic adsorbent, cesium silicate, and a strongly acidic adsorbent, 40% sulfuric acid impregnated silica gel (SA·SG), in the nonpolar solvent, hexane, and then subjected to chromatography on acid alumina. Application of the sample to cesium silicate in the low-polarity solvent hexane virtually assures the removal of trace residues of acidic compounds. Use of cesium silicate which has been activated at 130 °C resulted in poor recoveries of hepta- and octachloro isomers. The adsorbent should simply be purged of solvent under a stream of nitrogen after preparation and not oven activated.

The sulfuric acid impregnated silica gel (40% w/w) has been demonstrated in this laboratory and elsewhere (*51*) to strongly retain or undergo chemical reactions with a number of classes of compounds. A series of polynuclear aromatic hydrocarbons (PAHs) possessing two to four condensed rings was found in this laboratory to be effectively retained by this adsorbent. The adsorbent is also undoubtedly very effective in removing numerous types of compounds by reactions of dehydration, acid-catalyzed condensations, and oxidation as demonstrated by the complete charring and polymerization of tissue extracts applied to this material. Colored bands of adsorbed materials are normally observed on the SA·SG adsorbent following sample application in part II of this procedure. The reactivity of this adsorbent toward PAHs is complementary to the activated-carbon adsorbent which strongly adsorbs certain PAHs which are subsequently recovered with the PCDDs, PCDFs, and non-ortho PCBs. Because polynuclear aromatic hydrocarbons will elute from alumina under the solvent conditions employed in the subsequent step involving alumina chromatography, it is important that PAHs be removed prior to this step. In some environmental samples, especially sediments, high concentrations of PAHs were frequently encountered.

The final step of the enrichment procedure, alumina chromatography, is designed primarily to separate PCDDs, PCDFs, and non-ortho PCBs from polychlorinated naphthalenes (PCNs), trace residuals of PCB isomers, and other polychlorinated aromatic compounds. In addition to PCDDs, PCDFs, and non-ortho PCBs the only classes of compounds which have been shown in this laboratory and elsewhere (*46*) to be recovered from the carbon are PCNs, polychlorinated biphenylenes, and certain polychlorinated PAHs. The alumina chromatography removes the large majority of the 75 possible PCN isomers, but four to six penta- and hexachloronaphthalenes are partially recovered with the PCDDs, PCDFs, and non-ortho PCBs. Use of basic alumina (190 °C activated) requires higher concentrations of methylene chloride to recover PCDDs and PCDFs.

In-House and Extralaboratory Evaluations and Validation Studies. The following studies and evaluations were made: (a) determinations of the mean recoveries of a series of representative compounds of the three chemical groups at selected concentrations, (b) determinations of the coefficient of variation associated with each set of recovery data, (c) estimation of the lower limit of detection and determination of the various congener groups or individual components in a variety of sample types, (d) evaluation of the degrees of interference posed by seven series of polychlorinated aromatic compounds which represent the greatest threat of producing false-positive data, and (e) determination of the success rate for completed analyses of approximately 200 environmental samples.

Recovery Studies. Initial recovery studies were performed by using an abbreviated procedure which did not incorporate either the silica gel in part I or the alumina chromatography in part II. This procedure was highly effective for the determination of PCDDs, PCDFs, and non-ortho PCBs in biological materials. The major disadvantage of this abbreviated procedure appeared to be the inclusion of a large number of polychlorinated PAHs such as PCNs in the analyte. Nevertheless, an abbreviated procedure excluding alumina chromatography has been successfully used in the analyses of over 30 environmental samples. PCNs were the most significant cocontaminant observed but did not interfere in the deter-

Table I. Recoveries of Selected PCDDs and PCDFs in Salmon Oil from Abbreviated Procedure: Potassium Silicate, Carbon-Glass Fibers, Cesium Silicate, and Sulfuric Acid-Silica Gel[a]

amount of sample	recoveries of selected compounds: 2,3,6,8-Cl_4-furan	2,3,7,8-Cl_4-dioxin	1,2,4,7,8-Cl_5-furan	1,2,3,4,7,8-Cl_6-furan	1,2,3,4,6,8,9-Cl_7-furan	OCDD	OCDF
10 ng each in 10 g of oil	109 [1]	115 [1]	115 [1]	113 [1]	117 [1]	86 [1]	79 [1]

amount of samples	recoveries of selected compounds: 2,3,7,8-Cl_4-furan	2,3,7,8-Cl_4-dioxin[b]	1,2,4,7,8-Cl_5-furan	1,2,4,6,7,9-Cl_6-furan	1,2,3,4,7,8-Cl_6-dioxin	1,2,3,4,6,8,9-Cl_7-furan	OCDD	OCDF
2.5 ng each in 10 g of oil	81 (9) [4]		70 (5) [4]	75 (5) [4]	82 (3) [4]	77 (5) [4]	87 (7) [4]	75 (5) [4]
10 ng each in 20 g of oil	102 (2) [4]		97 (3) [4]	84 (4) [4]	98 (2) [4]	87 (6) [4]	76 (3) [4]	74 (5) [4]
25 ng each in 10 g of oil	66 (2) [3]		80 (–) [2]	68 (3) [3]	76 (–) [2]	72 (8) [3]	66 (3) [3]	62 (14) [3]

[a] Recoveries were determined on a 12-m OV-17 WCOT glass column and electron capture detection (^{63}Ni) using helium at 50 cm/s and the following temperature program: 190 °C for 2 min, then 4 °C/min to 240 °C and hold 15 min. Numbers in parentheses are coefficients of variations. Numbers in brackets are the number of replicate samples analyzed. [b] 2,3,7,8-TCDD and 2,3,7,8-TCDF coeluted on the OV-17 column.

Table II. Recoveries of Selected PCDDs and PCDFs from Spiked Samples of Homogenized Whole Fish Using the *Unabbreviated Enrichment Procedure*

sample	recoveries of selected compounds: 2,3,6,8-Cl_4-PCDF	2,3,7,8-Cl_4-PCDF and PCDD	1,2,4,7,8-Cl_5-PCDF	1,2,4,6,7,9-Cl_6-PCDF	1,2,3,4,7,8-Cl_6-PCDD	1,2,3,4,6,7,9-Cl_7-PCDF	OCDD	OCDF
100 g of grass carp and 10 ng each of PCDD and PCDF (100 pptr)	81 (1) [4]	92 (3) [4]	94 (3) [4]	98 (6) [4]	104 (4) [4]	95 (8) [4]	99 (22) [4]	91 (16) [4]

sample	recoveries of selected compounds: [^{13}C]-2,3,7,8-TCDD	[^{37}Cl]-2,3,7,8-TCDF	[^{37}Cl]-1,2,7,8-TCDF	[^{37}Cl]-OCDD
samples spiked at 25–50 pptr	82 ± 27 [49]	58 ± 16 [11]	75 ± 18 [10]	83 ± 30 [18]

	recoveries of selected compounds[a]: Cl_4 PCDFs	Cl_5 PCDFs	Cl_6 PCDFs	Cl_7 PCDFs	OCDF	Cl_5 PCDD	Cl_6 PCDD	Cl_7 PCDD	Cl_4 biphenylene
fish spiked at 20 pptr	58 ± 10	64 ± 6	64 ± 7	63 ± 10	59	41	49	58	52
fish spiked at 100 pptr	52 ± 7	55 ± 4	53 ± 6	56 ± 4	52	84	60	51	59

[a] Reference 45.

minations of PCDDs and PCDFs. The recoveries of a series of PCDDs and PCDFs from spiked samples of salmon oil by using the abbreviated procedures are given in Table I. Analyses of spiked fish samples containing up to 20 g of oil were also carried out by GC/EC and showed very low levels (*equivalent to less than 50 pptr for the most prominent* components) of matrix components in the analytes (*49*).

Following incorporation of silica gel in part I and alumina in part II of the procedure, recoveries of a series of PCDDs and PCDFs from spiked whole fish samples were again determined (Table II). Recently, an independent evaluation of the enrichment procedure was carried out at the University of Umea laboratory and included the determinations of recoveries from spiked fish of a mixture of fourteen tetra-, five penta-, five hexa-, three hepta-, and one octachlorodibenzofurans, one penta-, one hexa-, and one heptachlorodibenzodioxins, and one tetrachlorobiphhenylene (*45*). Mean and standard deviations of the recoveries are presented herein to further support the effectiveness of the method for the congener groups.

Ortho-unsubstituted PCBs have been detected in about 90% of the environmental samples analyzed by this method. Only two sets of recovery determinations have been made for three representative non-ortho PCBs spiked at 100 ppb: 3,4,3′,4′-tetrachloro (38 and 57%), 3,4,5,3′,4′-pentachloro (43 and 47%), and 3,4,5,3′,4′,5′-hexachloro (54 and 59%).

The demonstration of the effectiveness of recovery of a large *selection of* PCDD *and* PCDF *isomers, in particular those* tetra-, penta-, and hexachloro isomers possessing the critical 2,3,7,8-chlorine substitution pattern, is especially important to defining the comprehensiveness and applicability of the method. The recoveries of all the isomers studied are generally comparable and no particular isomer or group of isomers appear to be selectively excluded by the enrichment procedure.

In addition to the recovery data derived from spiked samples as part of the validation studies, a substantial collection of recovery data was also generated for the four major components of the marker compounds which were added to each sample prior to the enrichment process. The marker compounds, [UL-^{13}C]-2,3,7,8-TCDD, [UL-^{37}Cl]-OCDD, and a mixture of six [UL-^{37}Cl]-TCDFs including [^{37}Cl]-1,2,7,8- and [^{37}Cl]-2,3,7,8-TCDFs as the major components, were routinely incorporated into each sample at levels of 50, 50, 25, and 25 pptr, respectively. Although the range of recovery data values

Table III. Relative Recoveries of Tetrachlorodibenzo-*p*-dioxins from the Unabbreviated Enrichment Procedure[a]

GC/MS peak no.	TCDD isomer	rel recovery	GC/MS peak no.	TCDD isomer	rel recovery
1	1368	1.20	8	1234, 1237, 1238, 1246, 1249	1.45
2	1379	1.27	9	1236, 1279	1.47
3	1378	1.57	10	1278, 1469	1.35
4	1369, 1247, 1248	1.47	11	1239	1.39
5	1268	2.13	12	1269	1.39
6	1478	1.30	13	1267	2.85
7	2378	1.00	14	1289	3.62

[a] A total of approximately 2 ng of TCDDs was applied i.d. the enrichment procedure. Determination was made on a 60 m × 0.25 mm i.d. SP2330 (Supelco, Inc.) capillary column under MID-EI mass spectrometric conditions: temperature, 200 °C for 1 min, then to 250 °C at 5 °C/min and hold; He carrier gas.

for these marker compounds generally reflects the reduced precision of GC/MS/DS quantitation of trace analytes using the external standard technique, the determinations of the recoveries of the marker compounds in these samples performed over nearly a 3-year period provide a practical measure of the performance of the enrichment procedure and the overall analytical method (Table II). The average recoveries observed for the marker compounds over this extended period were found to be consistently satisfactory with the exception of that of [^{37}Cl]-2,3,7,8-TCDF which in early studies was observed to be uniformly low in comparison with those of the other marker compounds, most conspicuously with those of the five other [^{37}Cl]-TCDFs. A reexamination of the elution profile of 2,3,7,8-TCDF from alumina suggested that this step could be the sources of the problem; 2,3,7,8-TCDF eluted very close to the collection cutoff point. The addition of 5 mL to the collection volume increased the recovery of [^{37}Cl]-2,3,7,8-TCDF to levels comparable with those of the other marker compounds.

Determinations of background levels of PCDDs, PCDFs, and non-ortho PCBs were routinely made as part of the quality control protocol. Procedureal blanks and samples of uncontaminated laboratory-reared fish, each spiked with the marker compounds, were incorporated at a frequency of about 20% within all sample sets. Analyses of these control samples served to define the background level for sample sets and to diagnose possible residue carry-over among samples. Of 14 procedural blanks, 1 produced a positive determination for 2,3,7,8-TCDD at 1.6 pptr, 7 were positive for OCDD (1, 5, 7, 8, 8, 10, and 11 pptr), 1 was positive for a 2,3,7,8-TCDF at 2 pptr, and 2 were positive for OCDF at 0.5 and 1.4 pptr. All other results for the 10 congener groups (total of 140 determinations) in these procedural blanks were negative and were characterized by an average lower limit of detection of approximately 2 pptr. Of 11 analyses of samples of laboratory-reared grass carp, 7 produced positive determinations for OCDD (5, 5, 5, 7, 18, 24, and 39 pptr), 7 were positive for 2,3,7,8-TCDF (1, 1.5, 2, 3, 3, 3, and 6 pptr), 1 was positive for a PnCDF at 4 pptr, 1 for a HCDF at 2 pptr, 3 for a HpCDF (1, 1, and 2 pptr), and 5 were positive for OCDF (1, 1, 2, 3, and 15 pptr). The remainder of the 110 determinations of PCDDs and PCDFs in these control fish were negative. The average limit of detection was approximately 2 pptr. Non-ortho PCBs were not observed in these control samples, and the average limit of detection for these compounds was approximately 5 pptr. In one series of control samples of laboratory-reared trout, a number of PCDF isomers were repeatedly detected at 10–20 pptr levels. These compounds were later identified as trace contaminants in the commercial fish feed used in the rearing.

Overall, background levels of PCDDs, PCDFs, and non-ortho PCBs were negligible, especially for those isomers possessing the 2,3,7,8-substitution pattern. Octachlorodibenzo-*p*-dioxin appears to be a common trace environmental contaminant, being detected in more than 50% of the fish samples at levels significantly above those observed in the procedural blanks.

Although repeated analyses of procedural blanks between sample sets established a nondetectable level of carry-over between biological samples containing widely varying concentrations of PCDDs, PCDFs, and non-ortho PCBs, sample cross-contamination (from a carbon column) was observed to result from certain types of samples containing abnormally high levels of these contaminants. The samples causing cross-contamination were pond and river sediments and a sample of Aroclor 1260, all containing relatively high concentrations of PCDFs. Carry-over of PCDFs was readily demonstrated to result from reuse of the carbon columns and was observed in samples of fish which were processed on the same carbon column used for the highly contaminated samples. The degree of carry-over appeared to be on the order of 0.1%. In general, procedural blanks should be incorporated in sample sets at a frequency which will permit early detection of carry-over problems and should be included immediately following samples suspected of containing abnormally high concentrations of PCDDs, PCDFs, and non-ortho PCBs. Particularly in the case of sediment samples, high levels of other types of contaminants are routinely encountered, especially polynuclear aromatic hydrocarbons, and saturation of the carbon adsorbent with these substances may contribute to the problem of carry-over of PCDDs and PCDFs. In two cases of gross contamination of the carbon adsorbent, repeated washings of the column did not completely eliminate the problem, and the columns were replaced.

A satisfactory and reproducible level of recovery for 2,3,7,8-TCDD having been established, the recoveries of the other 21 TCDD isomers were examined. The mass chromatograms of a mixture of the 22 TCDD isomers (mixture provided by Dr. H. R. Buser, Swiss Federal Research Station, Wadenswil, Switzerland) before and after having been subjected to the enrichment procedure are presented in Figure 4. The relative recovery data, normalized to the recovery of 2,3,7,8-TCDD, are given in Table III. These data, although not rigorously demonstrative of satisfactory recoveries for each of the other 21 isomers, do establish that most of these isomers were effectively recovered by the procedure. In fact, in this experiment all other isomers or groups of isomers were apparently recovered more efficiently than was 2,3,7,8-TCDD. The abnormally high calculated recoveries of the 1,2,6,8-, 1,2,6,7-, and 1,2,8,9-TCDDs, each a minor component of the mixture, are attributed to the disproportionate influence of variations in instrumental sensitivity on analyte response near the limit of quantitation.

Probably the most useful piece of information derived from an examination of the determinations of the marker compounds in the hundreds of samples was the fact that the success rate for analyzability of the samples was better than 99% and that the minimum level of detection consistently

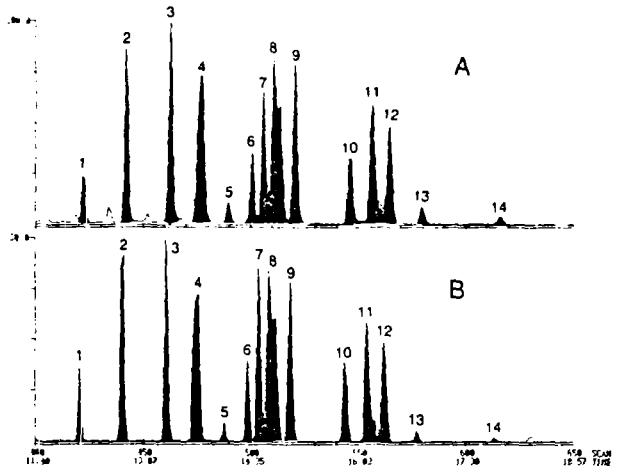

Figure 4. GC/MS-MID electron impact ion chromatograms of 22 TCDD isomers: (A) following application of enrichment procedure; (B) before enrichment.

fell in the range of 1–10 pptr with an average value of less than 5 pptr. Samples and controls were routinely spiked at the 20–50 pptr level with each of the marker compounds. In all cases, as indicated by the positive and uniform responses of the marker compounds in each of the analytes, GC/MS analyses of PCDDs and PCDFs at low parts-per-trillion levels were consistently attainable. Estimates of the lower limit of detection (LOD) for TCDDs, TCDFs, and OCDD were made by extrapolation from the observed signal-to-noise value for each of the marker compounds (internal standards) to the concentration corresponding to a signal-to-noise value of 3. Estimates of LOD require comparisons of the noise levels in the MID scans of each group of compounds and appropriate calibrations of the internal standards.

Confirmation of PCDDs, PCDFs, and non-ortho PCBs becomes increasingly difficult at levels approaching the limit of detection due particularly to increased variations in the relative intensities of the isotopic components of the molecular ions. The requirement of the correct isotopic abundance ratios for the molecular ions in determinations of PCDDs and PCDFs at low parts-per-trillion levels was usually the most difficult criterion to meet once sufficient instrumental sensitivity was attained. Nevertheless, over 50 separate confirmations were made of PCDD and PCDF residues present at less than 5 pptr. The criteria for the confirmation of any PCDD, PCDF, or non-ortho PCB of unspecified substitution pattern follow: (1) signal-to-noise ratio of ≥3; (2) correct nominal molecular mass; (3) coincidental maxima of three or more selected ion scans of individual members of the molecular isotopic cluster; and (4) chlorine isotope ratios within 10% of the correct values for three to six members of the molecular ion cluster.

The effectiveness of routine monitoring of the fragment ions resulting from characteristic loss of COCl from PCDDs and PCDFs was investigated and determined to be marginal for determinations at part-per-trillion levels due to the relatively weak signals for these ions. The criteria for confirmation of specific isomers also include a requirement of demonstrating the correct and unique relative retention time within 2–4 parts in 1000. For example, 2,3,7,8-TCDD is sufficiently resolved from the 21 other TCDD isomers on both a Silar 10C (*31*) and a SP2330 (Supelco, Inc.) (*52*) capillary column to enable easy determination of acceptable limits for the variation in retention time of this isomer relative to that of the isotopic marker [UL-^{13}C]-2,3,7,8-TCDD. The retention time of 2,3,7,8-TCDD on the DB-5 column was also found to be unique, although partial overlap with the 1,2,3,7- and 1,2,3,8-isomers indicated that their presence could be obscuring but would not produce a false-positive determination. The variation in the retention time of 2,3,7,8-TCDD relative to that of [^{13}C]-2,3,7,8-TCDD on the DB-5 column was observed in numerous analyses of standard mixtures of the two compounds and found to be within 2 parts in 1000. All confirmations of 2,3,7,8-TCDD in samples analyzed by this procedure met this requirement and were often repeated on a Silar 10C column. Samples of particular importance were independently analyzed by other laboratories using complementary techniques such as high-resolution mass spectrometry or atmospheric-pressure chemical ionization mass spectrometry (*53*). Over 20 samples analyzed in this laboratory for PCDDs and PCDFs were subjected to independent analyses in other laboratories, including those of H. R. Buser (Switzerland Federal Research Station, Wadenswil, Switzerland) (*54*), Ronald Mitchum (National Center for Toxicological Research, Jefferson, AR) (*55*), Michael Gross (University of Nebraska, Lincoln, NE) (*55*), Robert Harless (USEPA, Research Triangle Park, NC), David Firestone (U.S. Food and Drug Administration, Division of Chemistry and Physics, Washington, DC) (*56*), John Ryan (Health and Welfare Canada, Food Division, Ottawa, Canada) (*57*), Patrick O'Keefe (New York State Department of Health, Albany, NY) (*26*), and Christopher Rappe (University of Umea, Umea, Sweden) (Table IV). The Columbia laboratory also participated in three interlaboratory studies of the effectiveness of different methods for the determination of 2,3,7,8-TCDD in fish. The agreement in both identification and quantitation between the results from this laboratory and those of the other laboratories was consistently good, and no false-positive results were indicated in any of the determinations made with this procedure (Table IV). In the majority of interlaboratory studies, the comparisons involved only determinations of 2,3,7,8-TCDD.

Evaluation of Potential for Interference from Cocontaminants. Determinations of PCDDs, PCDFs, and non-ortho PCBs in environmental samples at levels below 1 pptr are particularly susceptible to interferences and possible false-positive results as a consequence of the likely occurrence of a large variety of polychlorinated aromatic cocontaminants and because full-scan mass spectrometric analyses are usually unattainable. More than a dozen families of such compounds are recognized as potential interferences in these types of analyses (*35*, *58*), including DDE and DDT and polychlorinated members of the following compounds: biphenyl (*59*), methoxybiphenyls (*60*), hydroxybiphenyls, diphenyl ether (*61*), methoxydiphenyl ethers, hydroxydiphenyl ethers (*62*), benzyl phenyl ether (*63*), naphthalene, biphenylene, phenylbenzoquinone (*64*), xanthene, and bis(phenoxy)methane (*65*). Most of these families of compounds have the potential to interfere with and produce false-positive results in determinations of PCDDs and PCDFs even in HRMS (*35*). The problem of interferences in determinations of PCDDs and PCDFs has been rigorously addressed experimentally in only a few publications (*66*), and in these was limited to a small proportion of the numerous families of potential interferences. Routinely, conclusions in regard to the potential for interferences in analytical procedures for PCDDs and PCDFs are made by inference from observations of the effectiveness of separation of comparable amounts of these interfering compounds from PCDDs and PCDFs, often with a relatively small number of isomers of these two families. For example, alumina has been shown to effectively separate PCBs from certain PCDD isomers (*67*). A more appropriate evaluation should include a large number of isomers of and a large excess concentration (10^4–10^6) of the potential interference relative to that of PCDDs or PCDFs.

As part of the validation of this procedure an evaluation was made of the degrees of interferences produced by seven

Table IV. Results of Interlaboratory Studies and Comparisons of the Determination of 2,3,7,8-TCDD in Fish and Birds

		levels of 2,3,7,8-TCDD reported (pg/g) at different laboratories							
study	CNFRL	no. 1	no. 2	no. 3	no. 4	no. 5	no. 6	no. 7	reported av
USFDA[a]									
sample 1	9					6	5		
sample 2	47	67			77	89	67		
sample 3	22	25			57	42	34		
sample 4	117	113		*b*	128	99	188		
sample 5	56	45	*b*	*b*	38	53	*c*		
sample 6	96	100	*b*	*b*	107	199	178		*b*
H&WC/USFDA[d]									
sample 7	58	104	58	49, 58	<5	72	70	60	61
sample 8	<1	<10	<1	<2, <2	<5	<2	<5	37	3.6
sample 9	34	35	37	23, 32	51	25	33	26	30
sample 10	38	45	33	19, 31	55	32	27	32	32
USEPA[e]									
sample 11	37	52	45	55					
sample 12	36	39							
sample 13	19	15	25						
sample 14	<1	<9	<5	<25					

Independent Laboratories

	CNFRL	Swiss Fed Res[f]	Nat Center Tox Res[g]	Health & Wel Can.[h]
herring gull, Lake Huron	160	165		132
gull egg, Detroit River	70	75		80
carp, Lake Huron	22, 27	29	10	
carp, Lake Erie	<1	5	<10	
lake trout, Lake Ontario	56, 58		54	
ocean herring, control	<1		<10	
lake trout, Lake Huron	39		32	
r.b. trout, Lake Ontario	38		31	
carp, Saginaw Bay	94		75	
carp, Tittabawassee R., MI	81		65	

[a] Reference 49. [b] Samples were not analyzed due to large amounts of materials in analyte. [c] Sample was lost. [d] Reference 50. [e] Reference 48. [f] HRGC/MS EI. [g] HRGC/MS API. [h] HRGC/HRMS EI.

families of polyhalogenated aromatic compounds (*35*). Included in the study were selected isomers of polychlorinated biphenyls (PCBs), naphthalenes (PCNs), diphenyl ethers (PCDPEs), methoxybiphenyls (MEO-PCBs), methoxydiphenyl ethers (MEO-PCDPEs), hydroxybiphenyls (HO-PCBs), and hydroxydiphenyl ethers (HO-PCDPEs). The study establishes an upper limit to the level of interference for each of these individual compounds. The results demonstrate the ability of the procedure to effectively eliminate interferences from all but a small number of PCN isomers and all PCBs (except non-ortho) present at concentrations of 10^6-fold in excess of those of the PCDDs and PCDFs. Levels of PCBs 100 000–500 000 times those of PCDDs and PCDFs were consistently observed in environmental samples analyzed in this laboratory (*68*), but PCB isomers other than the non-ortho PCBs have not been observed in the analyses for PCDDs and PCDFs. Furthermore, the results suggest that the procedure is not susceptible to interference from 10 000-fold excesses of the other five families of compounds. About six PCN isomers are recovered by the procedure and are commonly observed in environmental samples but do not produce false-positive determinations. Rarely, interference in the quantitation procedure due to partial overlap of a Cl_6 PCN isomer with the marker compound, [UL-^{13}C]-2,3,7,8-TCDD, is encountered. The effective elimination of numerous other organochlorine compounds, such as DDE, known to be present in many of the fish samples which were analyzed by this procedure has been demonstrated by full-scan MS analyses.

This procedure also recovers isomers of polychlorinated biphenylenes. A large number of isomers of polychlorinated biphenylenes were identified in this laboratory in a sample of soot produced during an electrical accident involving the pyrolysis of PCBs in a state office building in Binghamton, NY, in 1982 (*26*, *69*).

The only other group of polychlorinated aromatic compounds apparently observed in a small percentage of samples were the nonachloromethoxydiphenyl ethers. These compounds, of which there are three possible isomers, were tentatively identified in three fish samples, from Saginaw Bay (*35*, *68*), the Housatonic River, and Chesapeake Bay. The presence of these cocontaminants in the analyte contrasts with studies of interferences which indicate that chlorinated methoxydiphenyl ethers would readily be separated from PCDDs, PCDFs, and non-ortho PCBs.

The presence of polychlorinated diphenyl ethers (PCDPEs) in the analyte can be especially problematic because these compounds often undergo fragmentation during electron impact MS by loss of two chlorines to produce mass spectra which are identical with those of PCDFs below the molecular ion of the diphenyl ether. Furthermore, the elution window of PCDPE congeners have been observed in this laboratory to overlap that of PCDF congeners possessing two less chlorine substituents, greatly increasing the possibility for false-positive determinations from GC/MS-MIM analyses. Monitoring of masses of the molecular ions of the PCDPEs, if practical, can essentially eliminate this possibility.

The susceptibility to interferences of these types of analyses is demonstrated by the results of an interlaboratory study conducted by the USFDA (*56*) of the effectiveness of six different enrichment procedures (for 2,3,7,8-TCDD) performed by six independent laboratories (see Table IV). The enriched samples were all returned to the USFDA laboratory for rigorous analysis. Of the seven sets of analytical results only two,

Table V. Precision of Quantitation Using Internal Standards in GC/MS and GC/EC Analyses Before and After the Enrichment Procedure

	before enrichment procedure			after enrichment procedure		
compd[a]	mean response by GC/MS[b]	% std dev by GC/MS[b]	% std dev by GC/EC[c]	% std dev by GC/MS[b]	% rel recovery by GC/MS[b]	% rel recovery by GC/EC[c]
2468 F	1.39	6	8	12	97	109
1248 F	0.54	17	5	8	96	113
2368 F	1.40	7	2	14	97	128
1278 F	0.05		2		160	129
2378 F	1.05	7		5	85	
1234 D	0.92	15	4	19	140	127
2378 D	1.36	19	10	9	80	107
12478 F + 13478 F	5.63	10	2	15	109	126
12348 F	1.60	9	4	16	113	150
124689 F	1.29	9	5	17	133	137
234678 F	1.18	5	5	17	143	150
123478 D	0.80	8	7	15	153	141
1234689 F	0.97	11	4	20	135	157
1234678 D	0.42	12	8	26	195	159
OCDD	0.31	26	7	36	177	114
OCDF	0.44	27	7	30	164	114
[^{37}Cl]-1248 F	0.18	5		5	117	
[^{37}Cl]-1278 F	0.88	10		13	103	
[^{37}Cl]-2378 F	0.97	6		14	78	
[^{13}C]-2378 D	1.00				100	
[^{37}Cl]-OCDD	0.66	18		18	115	
mean		11.9	5.1	16.3		
mean, excluding Cl_8		9.8	4.9	14.1		

[a] F = PCDF, D = PCDD. [b] [^{13}C]-2,3,7,8-TCDD used as reference compound. [c] 2,3,7,8-TCDF used as reference compound.

including that generated by this laboratory, were judged to be uncompromised by the presence of significant levels of coextracted or interfering substances. In fact, the presence of excessive amounts of superfluous substances in a number of the analytes prevented the determination of TCDD in 5 of the 32 samples and apparently produced positive interferences in 3 fortified samples, as indicated by quantitative results which were significantly greater than the levels of fortification.

Quantitation Procedures. Quantitations of 2,3,7,8-TCDD, 2,3,7,8-TCDF, and OCDD are made directly by comparison of the integrated responses of the native compounds with those of the isotopically enriched marker compounds. Calibration is made by analysis of known amounts of the isotopic marker compound and an authentic quantitative standard of the native material under those GC/MS conditions used in analysis of samples.

During the first 2 years of use of this procedure, quantitations of other PCDDs, PCDFs, and non-ortho PCBs were made by the external standard technique using mixtures of approximately 12 compounds. Toward the latter half of 1982, the quantitations of these compounds were performed using the three major isotopic marker compounds as internal quantitation standards for all congeners. Usually [^{37}Cl]-OCDD was used for quantitation of OCDD and OCDF, and [^{13}C]-2,3,7,8-TCDD and [^{37}Cl]-2,3,7,8-TCDF were used for quantitations of all other PCDDs, PCDFs, and non-ortho PCBs. Average relative response factors for the various congener groups were determined by GC/MS analyses of mixtures of the isotopic marker compounds and a series of 20 synthesized PCDDs, PCDFs, and non-ortho PCB isomers.

An attempt was made to determine the suitability, in terms of accuracy and precision, of quantitations of all congener groups using the internal standards (isotopic marker compounds). The experiment involved GC/MS-MIM and GC/EC analyses (4 replicates each) of a mixture of 17 native PCDDs and PCDFs and the 5 isotopically enriched marker compounds. This mixture was subsequently subjected to the enrichment procedure (5 replicates) and analyzed again by GC/MS-MIM and by GC/EC. The mean and standard deviations of the integrated responses of all compounds relative to that of [^{13}C]-2,3,7,8-TCDD were determined by GC/MS, and 2,3,7,8-TCDF was used as the internal standard in GC/EC analyses (Table V). The level of variation as measured by standard deviation for GC/MS quantitations using the internal standard was twice that determined for the GC/EC analyses. The data indicate that GC/MS quantitations using TCDD or TCDF as an internal standard were significantly more precise for tetrachloro through heptachloro congeners than for OCDD and OCDF. In contrast, no such disproportionate trends in precision were observed in the GC/EC analyses. The large variations associated with OCDD and OCDF are believed to be in part a consequence of GC/MS instrumental problems which were being experienced at the time and not necessarily characteristic of these types of analyses. Analyses of the mixture following application of the enrichment procedure show that the mean standard deviation is increased but comparable to instrumental variation. Nevertheless, the results indicate an acceptable level of precision for GC/MS quantitations of Cl_4 through Cl_7 congeners using a TCDD or TCDF as an internal standard in samples subjected to the enrichment procedure.

Determinations of PCDDs, PCDFs, and non-ortho PCBs were routinely carried out in the electron impact GC/MS mode. The GC/MS-EI technique, in contrast to negative ion chemical ionization analysis, exhibits comparable sensitivity for the broad range of congeners and permits identification and quantitation of all components in a single analysis. Negative ion chemical ionization GC/MS (GC/MS-NICI) has been observed in this laboratory and elsewhere (70) to exhibit a markedly enhanced sensitivity to PCDFs relative to PCDDs and, generally, to the higher relative to the lower chlorinated congeners of both groups. The ability to determine tetrachlorodioxins and tetrachlorobiphenyls in particular suffers

under GC/MS-NICI, and consequently this technique is unacceptable for complete determination of PCDDs, PCDFs, and non-ortho PCBs at part-per-trillion levels. On the other hand, GC/MS-NICI is much less sensitive to background (especially column bleed and hydrocarbons) or cocontaminant substances and invariably yielded more easily interpretable data.

Efficiency of Extraction. The implicit assumption in quantitations using the internal standards incorporated at the beginning of the procedure is that the behavior of an isotopically enriched compound will be identical with that of the native compound present in sample. This assumption is generally endorsed for all enrichment processes except that of the extraction of residues from the sample matrix. The consideration of extractability of bioincorporated contaminants from biological samples or sorbed residues from soils or combustion products is particularly important in studies of PCDDs and PCDFs. Studies of the biochemistry of 2,3,7,8-TCDD and related compounds in mammalian systems (*71*) have established that these compounds exhibit high specific binding affinities for a hepatic cytosol protein; consequently, extraction of some PCDDs, PCDFs, and non-ortho PCBs from biological samples may involve more than the liberation of these residues from solution in fatty deposits. No studies have been reported of the efficiency of extract of bioincorporated PCDDs, PCDFs, or non-ortho PCBs. On the other hand, comparisons of the results of interlaboratory studies (Table IV) involving a wide variety of extraction procedures used for identical samples of fish containing bioincorporated 2,3,7,8-TCDD have provided a reasonable measure of the extractability of this substance from fish tissue. The results of these studies suggest that the neutral column extraction employed in this procedure is essentially equivalent in efficiency to extractions involving complete digestion of the tissue with concentrated aqueous base or acid. Such digestions are expected to denature and hydrolyze all proteins and to effectively liberate all intact TCDD residues. Referring to Table IV, laboratory no. 1 in the USFDA study employed digestion with concentrated HCl; in the H&WC/USFDA study, laboratory no. 3 employed digestion with KOH, and laboratory no. 7 employed digestion with HCl. Assuming that 2,3,7,8-TCDD is as strongly bound in these samples of fish tissue as are any other PCDD, PCDF, or non-ortho PCB, the neutral extraction procedure is expected to effectively recover all intact residues of these compounds. The effectiveness of the neutral extraction could be species dependent and cannot be extended to other animal systems without similar studies being made. Our rationale for addition of the internal standards to the samples at the beginning of the extraction process rather than before homogenization and mixing of the sample with sodium sulfate was that equilibration of the native residues with the internal standards could not be easily attained in the latter step. Consequently, losses in the homogenization and drying step are not included in the internal standard quantitation procedure.

The strong adsorptive interaction of PCDDs, PCDFs, and non-ortho PCBs with carbonaceous materials has been studied in depth (*37*), and studies of fly ash containing these compounds have demonstrated that exhaustive extraction procedures are required (*72*). Consequently, a study was undertaken in this laboratory to determine the relative efficiencies of two methods of extraction of these compounds from Hudson River sediment samples (*73*). The neutral column extraction procedure was compared with a procedure (*72*) which has been demonstrated to be effective for the recovery of PCDDs from fly ash. Although the results of the comparison study were highly variable and no unambiguous measure of the relative efficiencies of the two procedures could be made, neither of the procedures was uniformly superior

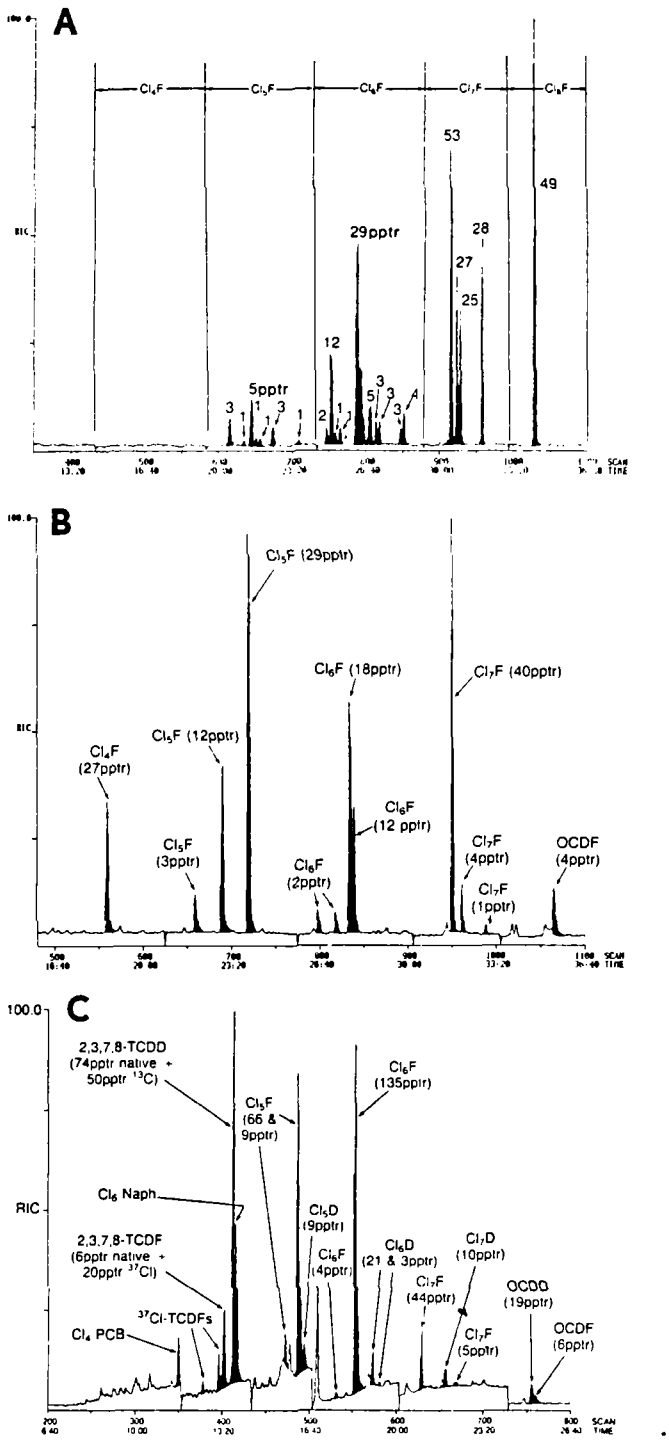

Figure 5. Representative analyses of environmental samples: (A) GC/NICI-MS-MID PCB contaminated soil from Fountain City, WI; (B) GC/NICI-MS-MID fish sample (carp) from Saginaw Bay at Bay City, MI; (C) GC/EI-MS-MID fish sample (carp) from the Niagara River at Ft. Niagara, NY.

to the other and appear to be roughly comparable in effectiveness. More definitive results are required from such studies before the efficacy of the column extraction procedure in analyses of soil and sediment samples can be established.

Applications to the Analyses of Environmental Samples. The procedure has been applied to the determination of PCDDs, PCDFs, and non-ortho PCBs in a wide range of sample types, primarily fresh-water fishes. The sample types which have been analyzed include about 12 species of fresh water fish (*55*, *68*) and three species of salt water fish (both whole body and fillet): snapping turtle fat (*54*), whole body

crayfish, approximately five species of fresh water mussels, whole body, muscle and eggs of three species of birds, Baltic seal fat (*54*), aquatic macroinvertebrates, commercial fish feeds, aquatic and terrestrial soils (*73*), soot from an office building fire involving PCBs and polychlorinate benzenes (*26*), a sample of Aroclor 1260, and failed transformer fluid from a waste disposal site. The large majority of these samples were taken from sites on the five Great Lakes and selected tributaries, the Ohio, Mississippi, Hudson, and Sacramento Rivers, 12 eastern seaboard rivers and estauries, and the Housatonic River in Massachusetts and Connecticut known to be contaminated with a wide range of persistent synthetic chemicals such as PCBs, organochlorine pesticides, and industrial wastes.

The total number of samples analyzed was approximately 200, not including over 50 control and procedural blank samples. Essentially all of the 250 analyses were judged to be successful according to the following criteria: (1) All marker compounds were detected in the analyte. (2) An acceptable limit of detection (usually less than 5 pptr) was achieved. (3) The levels and GC/MS properties of analyte components other than PCDDs, PCDFs, and non-ortho PCBs did not produce significant interferences. (4) The criteria for the determination of PCDDs, PCDFs, and non-ortho PCBs were met.

Representative multiple ion mass chromatograms of soil and fish samples are presented in Figure 5. These GC/MS determinations of PCDDs, PCDFs, and non-ortho PCBs in widely differing types of samples serve to exemplify the versatility of the procedure for such analyses. The GC/MS data were usually uncluttered by extraneous components, and interpretation of the data was routinely straightforward.

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Selectivity of Negative Ion Chemical Ionization Mass Spectrometry for Benzo[*a*]pyrene

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Gas chromatography/negative ion chemical ionization mass spectrometry (GC/NICIMS) was used as a selective and sensitive technique for the detection of benzo[*a*]pyrene (BaP). Under optimized conditions, the molecular anion, $M^{-\cdot}$, of BaP was more than 3 orders of magnitude more abundant than that of its isomer benzo[*e*]pyrene (BeP) using methane as the reagent gas. Quantities of BaP as low as 1 pg can easily be detected in the selected ion monitoring mode and the response vs. concentration was linear over a range of 3 orders of magnitude. The absolute sensitivity and the selectivity for detection were found to depend on the pressure and temperature in the ion source of the mass spectrometer. NICIMS was used for the quantitative determination of BaP, indeno[1,2,3-*cd*]pyrene, and benzo[*ghi*]perylene in a sample of petroleum crude oil as part of the process of certifying the oil as a Standard Reference Material.

Negative ion chemical ionization (NICI) mass spectra can be obtained from certain organic compounds by resonance capture of thermal electrons if the molecules have positive electron affinities, and if the internal energy of the molecular anion is less than the electron affinity of the neutral species. Usually the major species formed is the molecular anion, $M^{-\cdot}$, which often yields relatively large ion currents and little fragmentation. The selectivity of NICI over electron impact (EI) has been well established and this feature has permitted NICI to have wide applications over the past few years in the analysis of compounds such as polychlorinated biphenyls (*1*), dioxins (*1-3*), pesticides (*1*, *4*, *5*), and nitrated polycyclic aromatic hydrocarbons (*6*). Ilda and Dashima (*7*) recently reported the methane negative ion chemical ionization mass spectra of 21 polycyclic aromatic hydrocarbons (PAH). Oehme (*8*) determined PAH in air particulate matter using NICI. He used a mixture of methane and nitrous oxide as the reagent gas to promote ionization by electron capture and ion/molecule reactions and was able to differentiate isomeric PAH based on the relative abundances of various species formed. Zackett, Ciupek, and Cooks (*9*) used negative ion chemical ionization charge inversion mass spectrometry as a highly selective means for determining polycyclic aromatic hydrocarbons in a solvent refined coal.

We have used NICI mass spectrometry as a sensitive and selective technique for the quantitative determination of benzo[*a*]pyrene (BaP) in a sample of petroleum crude oil which is being certified as a Standard Reference Material (SRM). During the course of preliminary studies we have confirmed the large degree of selectivity for the detection of BaP over benzo[*e*]pyrene (BeP) noted by others (*7*, *8*). We have observed the molecular anion of BaP to be more than 1000 times more abundant than that of BeP under selected source conditions in the NICI mode using methane as the reagent gas. Our observations, reported here, show that the ion source pressure and temperature play an important role in the selectivity of detection for BaP. We have also observed excellent absolute sensitivity for the detection of BaP and are able to detect quantities as low as 1 pg in the selected ion monitoring mode.

EXPERIMENTAL SECTION

Negative ion chemical ionization mass spectra were recorded on a Hewlett-Packard 5985B quadrupole GC/MS system (Hewlett-Packard Co., Palo Alto, CA) with a dual EI/CI ion source and electronics capable of detecting negative ions. Chromatographic separations were carried out on a 30 m × 0.25 mm i.d. fused silica capillary column coated with a 0.25-μm film of a nonpolar liquid phase. Samples were injected in either the split or splitless modes with an injection port temperature of 300 °C and the column temperature was programmed from 200 to 300 °C at a rate of 4 °C/min. The column was interfaced directly to the ion source by inserting it through a 30 cm length of 0.16 cm o.d. stainless steel tubing. The stainless steel tubing also served as a conduit for introduction of the methane reagent gas (Matheson Ultra High Purity 99.97%) which was brought in coaxially with the capillary column. The pressure in the ion source was adjusted by varying the methane flow into the source via a flow controller. An ionization gauge, which was mounted approximately 15 cm from the source, was used to monitor the ion source manifold pressure. The pressure in the ion source itself was measured with a thermocouple gauge. Spectra were recored under conditions optimized empirically for the detection of BaP. The ion source was normally operated at 200 °C with a filament emission current of 300 μA and a primary electron beam energy of 60 eV. The mass spectrometer was calibrated in the NICI mode using ions at m/z 414, 452, and 633 from perfluorotributylamine and ions at m/z 233 and 235 from rhenium oxide generated by the filament. The ReO_3^- isotopes provide a good source of ions at low mass for tuning the mass spectrometer in the negative ion mode.

The PAH were obtained commercially: BaP (Community Bureau of Reference, BCR, Brussels, Belgium); BaP-d_{12} 98.6 atom % D (MSD Isotopes, St. Louis, MO); and BeP (Pfaltz and Bauer, Inc., Stamford, CT). The standards were analytical grade or higher and were used without further purification. Methylene chloride solutions of the PAH were prepared gravimetrically. The Wilmington crude oil sample was obtained from the Department of Energy and is one of the oils being stored in the EPA Repository

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ATTACHMENT 6

ANALYTICAL PROCEDURES FOR EXPLOSIVES IN SOILS

METHOD NO.: 8H

DATE: 4-21-83

EXPLOSIVES IN SOIL BY HPLC

I. APPLICATION: Determination of the following nitro-compounds in soil.

HMX	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
RDX	Hexahydro-1,3,5-trinitro-s-triazine
NB	Nitrobenzene
1,3-DNB	1,3-Dinitrobenzene
1,3,5-TNB	1,3,5-Trinitrobenzene
2,4-DNT	2,4-Dinitrotoluene
2,6-DNT	2,6-Dinitrotoluene
2,4,6-TNT	2,4,6-Trinitrotoluene
Tetryl	2,4,6-Trinitrophenylmethylnitramine

A. Tested Concentration Range:

HMX	0.376-188 ug/g
RDX	0.253-127 ug/g
NB	0.197-98.4 ug/g
1,3-DNB	0.242-121 ug/g
1,3,5-TNB	0.215-107 ug/g
2,4-DNT	0.240-120 ug/g
2,6-DNT	0.217-109 ug/g
2,4,6-TNT	0.301-151 ug/g
Tetryl	0.265-133 ug/g

B. Sensitivity: Peak height near the detection limit. (1 mm = 28 arbitrary units on the integrator readout.) Representative chromatogram near the detection limit can be found in Appendix I.

	Peak Height in mm at an Attenuation of 2^{-2}
HMX	12 mm for 0.754 ug/g
RDX	18 mm for 0.506 ug/g
NB	11 mm for 0.394 ug/g
1,3-DNB	23 mm for 0.485 ug/g
1,3,5-TNB	20 mm for 0.430 ug/g
2,4-DNT	16 mm for 0.480 ug/g
2,6-DNT	9 mm for 0.434 ug/g
2,4,6-TNT	19 mm for 0.602 ug/g
Tetryl	10 mm for 0.530 ug/g

C. Detection Limits:

HMX	0.376 ug/g
RDX	0.474 ug/g
NB	0.197 ug/g
1,3-DNB	0.242 ug/g
1,3,5-TNB	0.231 ug/g
2,4-DNT	0.240 ug/g
2,6-DNT	0.217 ug/g
2,4,6-TNT	0.301 ug/g
Tetryl	0.265 ug/g

D. Interferences:

1. Any compound that is extracted from soil that gives a retention time similar to the nitro-compounds and absorbs U.V. at 250 nm.

2. Millipore GFWP-01300 filter type GS pore size 0.22 micrometers dissolve in the solvent used.

3. Tetryl and 2-amino-4,6-dinitrotoluene coelute. If a tetryl peak is found in samples, pH adjustment is necessary to separate the peaks to determine which compound is present.

4. 2,4,6-Trinitrobenzaldehyde decomposes rapidly in water solution. Once the acetonitrile standard is made into mobile phase this becomes a problem.

E. Analysis Rate:

After instrument calibration, one analyst can analyze two samples in one hour. One analyst can conduct sample preparation at a rate of three samples per hour. One analyst doing both sample preparation and the HPLC analysis can run 16 samples in an 8-hour day.

II. CHEMISTRY:

A. Chemical Abstracts Service Registry Number:

HMX	2691-41-0
RDX	121-82-4
NB	98-95-3
1,3-DNB	99-65-01
1,3,5-TNB	99-35-4
2,4-DNT	121-14-2
2,6-DNT	606-20-2
2,4,6-TNT	118-96-7
Tetryl	479-45-8

B. Chemical Reactions:

1. RDX and HMX can undergo alkaline hydrolysis.

2. RDX and HMX degrade at temperatures greater than 80°C in an organic solvent.

C. Physical Properties:

	Formula	Mol. Wt.	M.P.(°C)	B.P.(°C)
HMX	$C_4H_8N_8O_8$	296.16	276	-
RDX	$C_3H_6N_6O_6$	222.12	205	-
NB	$C_6H_5NO_2$	123.11	6	211
1,3-DNB	$C_6H_4N_2O_4$	168.11	90	302
1,3,5-TNB	$C_6H_3N_3O_6$	213.11	122	315
2,4-DNT	$C_7H_6N_2O_4$	182.14	71	300 (decomposes)
2,6-DNT	$C_7H_6N_2O_4$	182.14	66	-
2,4,6-TNT	$C_7H_5N_3O_6$	227.13	82	240 (decomposes)
Tetryl	$C_7H_5N_5O_8$	287.15	131	187

III. APPARATUS:

A. Instrumentation: Perkin Elmer series 4 High Performance Liquid Chromatograph (HPLC) equipped with a Perkin Elmer ISS-100 Auto-Injector and Perkin Elmer variable wavelength detector LC-75. Hewlett Packard 3390 recording integrator in peak height mode was used to record the data output.

B. Parameters:

1. Column: Two columns are used in series, in the order listed.

   a. DuPont Permaphase^{R} ODS guard column.

   b. DuPont Zorbax^{R} ODS 4.6 mm i.d. x 25 cm HPLC column with a particle size of 5-6 microns.

2. Mobile Phase: The water/methanol ratio must be adjusted as described in the calibration Section V C to obtain optimum peak separation.

   44-50% water
   28-34% methanol
   22% acenotrile

3. Flow: 1.6 mL/min with a pressure of approximately 2860 psig.

4. Detector: 250 nm

5. Injection Volume: 50 uL

6. Retention Times:

	Minutes
HMX	3.38
RDX	4.21
NB	7.33
1,3 DNB	6.63
1,3,5-TNB	5.74
2,4-DNT	9.89
2,6-DNT	9.50
2,4,6-TNT	8.93
Tetryl	7.98

C. Hardware/Glassware:

1. Syringes: 25 uL, 50 uL, 100 uL, 250 uL, 5 mL gas tight syringe (Hamilton 1005 TEFLL)

2. Serum vials with crimp caps and Teflon-lined septa Nominal volume of 0.25 mL, 1 mL, 5 mL.

3. Pasteur pipettes and disposable micropipettes.

4. 13 mm stainless steel syringe filter holder (Rainin Instrument Co., Inc. #38-101)

C. Hardware/Glassware: (continued)

5. 13 mm x 0.5 micron fluorocarbon filter (Rainin Instrument Co., Inc. #38-103 Zefluor disc)

6. Whatman 10 mm glass microfiber prefilter

7. U.S. Sieve series 600 (30 mesh)

8. Aluminum foil pans

9. Liquid chromatograph column 1" o.d. x 12"

10. 2 mL, 3 mL, and 5 mL pipettes

D. Chemicals:

1. Acetonitrile, distilled in glass for HPLC use
2. Methanol, distilled in glass for HPLC use
3. Ethyl Ether, distilled in glass for HPLC use
4. Hexane, distilled in glass for HPLC use
5. ASTM Type II Water
6. SARMs for the nitro-compounds

IV. STANDARDS: All concentrations are based on a stock solution concentration of 2000 mg/L. Appropriate adjustments should be made if actual concentration varies from this figure.

A. Calibration Standards:

1. Stock Calibration Standards: Stock solutions containing approximately 2000 mg/L of a nitro-compound are prepared by accurately weighing 10 mg of a SARM into a 5 mL serum bottle and dissolving the nitro-compound in 5 mL of acetonitrile pipetted into the bottle. All compounds appear to be stable for 3 months.

2. Intermediate Calibration Standards: All compounds appear to be stable for 3 months.

1. Intermediate Calibration Standard A (high level): Add the following volumes of stock calibration standard and seal with a Teflon-lined septum cap. Store in the dark @ 0°-4°C. The resulting solution (5.8 mL) will have the concentrations indicated in the following table.

A. Calibration Standards: (continued)

Intermediate Calibration Standard A

Nitro-compound	Amt. (uL) of Stock Cal. Std. to add	Resulting conc. (ug/mL)
HMX	1000	345
RDX	600	207
NB	400	138
1,3-DNB	500	172
1,3,5-TNB	500	172
2,4-DNT	500	172
2,6-DNT	500	172
2,4,6-TNT	700	241
Tetryl	600	207
TNBA*	500	172

*2,4,6-Trinitrobenzaldehyde was originally included for certification. However, the compound is too unstable in water solutions to obtain reproducible certification data. It was included in this table as it affects the total volume used to calculate concentration of the other nitro-compounds.

b. Intermediate Calibration Standard B (low level):

Pipette 4.5 mL of acetonitrile into a 5-mL serum vial. Add 500 uL of Intermediate Calibration Standard A. Seal with a Teflon-lined septum cap and store in the dark @ 0-4°C. The resulting solution (5.0 mL) will have the concentrations indicated in the table below:

Intermediate Calibration Standard B

Nitro-Compound	Resulting conc. (ug/mL)
HMX	34.5
RDX	20.7
NB	13.8
1,3-DNB	17.2
1,3,5-TNB	17.2
2,4-DNT	17.2
2,6-DNT	17.2
2,4,6-TNT	24.1
Tetryl	20.7

A. Calibration Standards: (continued)

3. Working Calibration Standards: To a series of ten 5-mL serum vials, approximately one gram of prepared soil (see section V.B.) is accurately weighed into each vial. Using a syringe, the volumes of intermediate standard solutions indicated in the following table are injected onto soil. The serum vial is covered with a septum and shaken until the soil no longer looks wet (approximately 60 seconds). The septum is removed and the indicated amount (see Table below) of acetonitrile is pipetted onto the soil. The septum is replaced and the cap crimped on the vial. The sealed sample is blended on a vortex mixer for approximately 2-3 minutes. The sample is prepared via the procedure given in this method, to give the target concentrations in the following table.

WORKING CALIBRATION STANDARDS

Rel. Conc.	Amt. (uL) Intermed. Cal. Std. to Add		Amt. (mL) Aceto-Nitrile to Add	Resulting Concentration (ug/g)				
	A	B		HMX	2,4,6-TNT	Tetryl	1,3-DNB; 1,3,5-TNB; 2,6-DNT; 2,4-DNT	NB
0	0	0	2.0	0	0	0	0	0
0.1 X	-	12	2.0	0.414	0.289	0.248	0.206	0.166
0.2 X	-	24	2.0	0.828	0.578	0.497	0.413	0.331
0.5 X	6	-	2.0	2.07	0.145	1.42	1.03	0.828
1 X	12	-	2.0	4.14	2.89	2.48	2.06	1.66
2 X	24	-	2.0	8.28	5.78	4.97	4.13	3.31
5 X	60	-	2.0	20.7	14.5	14.2	10.3	8.28
10 X	120	-	1.9	41.4	28.9	24.8	20.6	16.6
25 X	240	-	1.8	82.8	57.8	49.7	41.3	33.1
50 X	600	-	1.4	207	145	142	103	82.8

B. Control Spikes: Control spikes are prepared in the same manner as the calibration standards.

V. PROCEDURE:

*NOTE THE FOLLOWING SAFETY PRECAUTIONS:

1. A 5-mL gas tight syringe (Hamilton 1005 TEFLL) is used, as the teflon/glass seal is less likely to cause an explosion than glass/glass.

2. The nitro-compounds are less reactive when wet, so every precaution should be taken to ensure that work areas are kept clean and that solutions are not left unattended and allowed to dry.

3. The filtering apparatus is immersed in a water bath and disassembled under water immediately after use. The danger here is solution getting dried on the threads of the filtering apparatus and detonating.

4. When preparing SARM stock standards from pure compounds which are stored in water, small aliquots are scooped onto a nylon or polyvinylidene chloride filter. The water is vacuum filtered off and an appropriate quantity of the "dried" material is weighed out for stock standard preparation. Any extra compound thus dried is disposed of.

5. Prior to working with explosives, it is advisable to discuss safety/handling/storage requirements with an explosives expert.

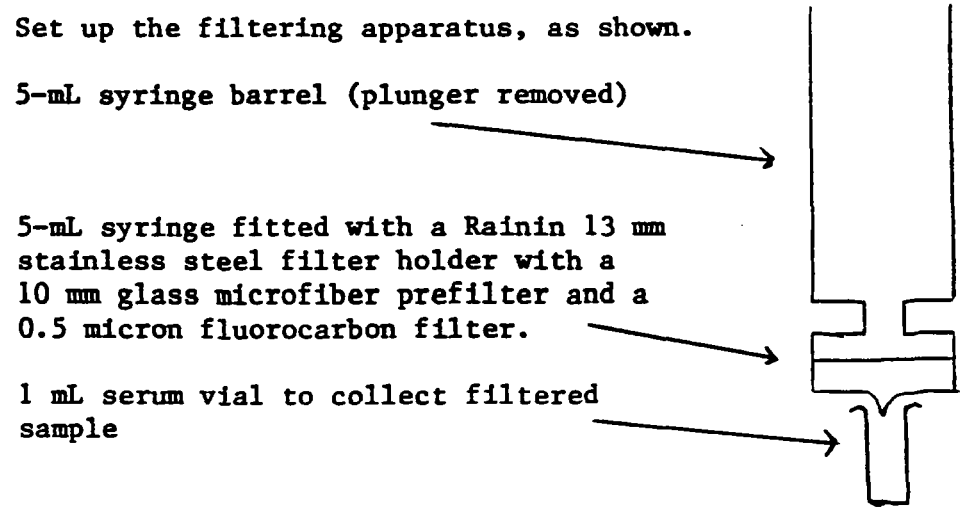
A. Sample Preparation: The soil sample is removed from the sample bottle and spread out in aluminum foil trays. The sample is air dried. The dried soil is screened through a US series 600 sieve (30 mesh). This screened sample is subsampled according to ASTM procedure D346. The moisture content is determined by ASTM Method D2216-71.

B. Extraction:

1. Accurately weigh 1 gram of prepared soil (see section V.A. above) into a 5-mL serum vial, and pipette 2 mL of acetonitrile onto the soil.

   Place a septum and cap on the vial, crimp into place, and shake the vial thoroughly on a vortex mixer for 2-3 minutes.

2. Set up the filtering apparatus, as shown.



V. PROCEDURE: (continued)

3. Prepare the sample for injection as follows:

   a. Pour the sample extract into the syringe.

   b. Place the plunger in the syringe and force at least 500 uL of the filtrate into a 1-mL serum vial.

   c. Using a disposable micropipette, accurately measure 200 uL of filtered extract into a 1-mL serum vial. Accurately measure 600 uL of a 33% methanol/67% water solution onto the filtered sample. This will produce 800 uL of extracted sample in mobile phase.

   d. Place a septum and cap on the vial and crimp into place. Shake the vial well to thoroughly mix. Store in the dark @ 0-4°C until ready to analyze.

4. For samples outside the calibration range, a smaller sample volume is extracted into 5-mL of acetonitrile.

   a. Accurately weigh 0.2 gram of prepared soil into a 5-mL serum vial, and pipette 5 mL of acetonitrile onto the soil. Place a septum and cap on the vial, crimp into place, and shake the vial thoroughly on a vortex mixer for 2-3 minutes.

   b. Prepare the sample for injection as follows:

      1) Pour the sample extract into the syringe.

      2) Place the plunger in the syringe and force at least 3 mL of the filtrate into a 5-mL serum vial.

      3) Using a disposable pipette, accurately measure 1 mL of filtered extract into a 5-mL serum vial. Accurately measure 3 mL of a 33% methanol/67% water solution onto the filtered sample. This will produce 4 mL of extracted sample in mobile phase.

         Alternately, the sample extract and methanol/water solution may be accurately weighed into a 5-mL serum vial. (1 mL $\approx$ 1 g)

4) Place a septum and cap on the vial and crimp into place. Shake the vial well to thoroughly mix. Store in the dark @ 0-4°C until ready to analyze.

c. If the solution prepared from the 0.2 g sample is still above the calibration range, make dilutions of the extract obtained in 4b(1) by taking an appropriate aliquot and adding mobile phase (e.g. 100 mg of acetonitrile sample extract in 20 mL mobile phase) to produce a solution within the calibration range of the instrument.

C. Instrument Calibration/Sample Analysis:

1. Using the auto-injector manufacturer's recommended procedure, introduce 50 uL of the 2X working calibration standard into the chromatographic system. Check the chromatogram to ensure separation of the nitrated toluenes and separation of the nitrobenzene and tetryl. If necessary, adjust the water/methanol ratio of the mobile phase until separate peaks are distinguished. As the column ages, less methanol is required. Generally, the column ages rapidly the first 24 hours, after which it is fairly stable.

2. Once good peak separation is obtained, introduce 50 uL of each working calibration standard and sample into the chromatographic system using the auto-injector manufacturer's recommended procedure.

VI. CALCULATIONS:

A. Sample Concentration (ug/g) $= \frac{(\text{peak ht.} - K) \times C \times E}{\text{slope} \times A \times B \times D}$

where:

K = y-intercept of the calibration curve regression line

slope = slope of the calibration curve regression line

$A = \frac{8 \text{ mL mobile phase}}{1 \text{ gram sample}}$ = a constant for this method.

Explanation: the instrument reads the total ug in the 50 uL aliquot of sample injected. This constant enables results to be interpreted as ug/g, as the calibration curve in ug/g is obtained by

$$\frac{2 \text{ mL acetonitrile to extract}}{1 \text{ gram calibration std. sample}} \times \frac{4 \text{ mL mobile phase}}{1 \text{ mL acetonitrile extract}}$$

VI. CALCULATIONS: (continued)

B = sample weight

C = mL acetonitrile used to extract sample

D = mL acetonitrile extract diluted into mobile phase

E = final volume in mL of mobile phase prepared for injection

NOTE: When samples are prepared the same as the calibration standards (1 gram extracted into 8 mL of mobile phase), the above calculation becomes:

$$\text{Sample Concentration (ug/g)} = \frac{(\text{Peak height} - K)}{\text{slope}}$$

B. All soils data must be reported on a moisture-free basis. Moisture content is determined by ASTM D2216-71. 100%-% Moisture = % solids.

$$\text{Concentration on a moisture free basis} = \frac{\text{analyte concentration}}{\% \text{ solids}} \times 100$$

VII. REFERENCES:

A. USATHAMA Method 2C Cyclotrimethylenetrinitramine (RDX) in Soil and Sediment Samples, 12-3-80.

B. USATHAMA Method 8H Explosives in Water by HPLC, 12-27-82.

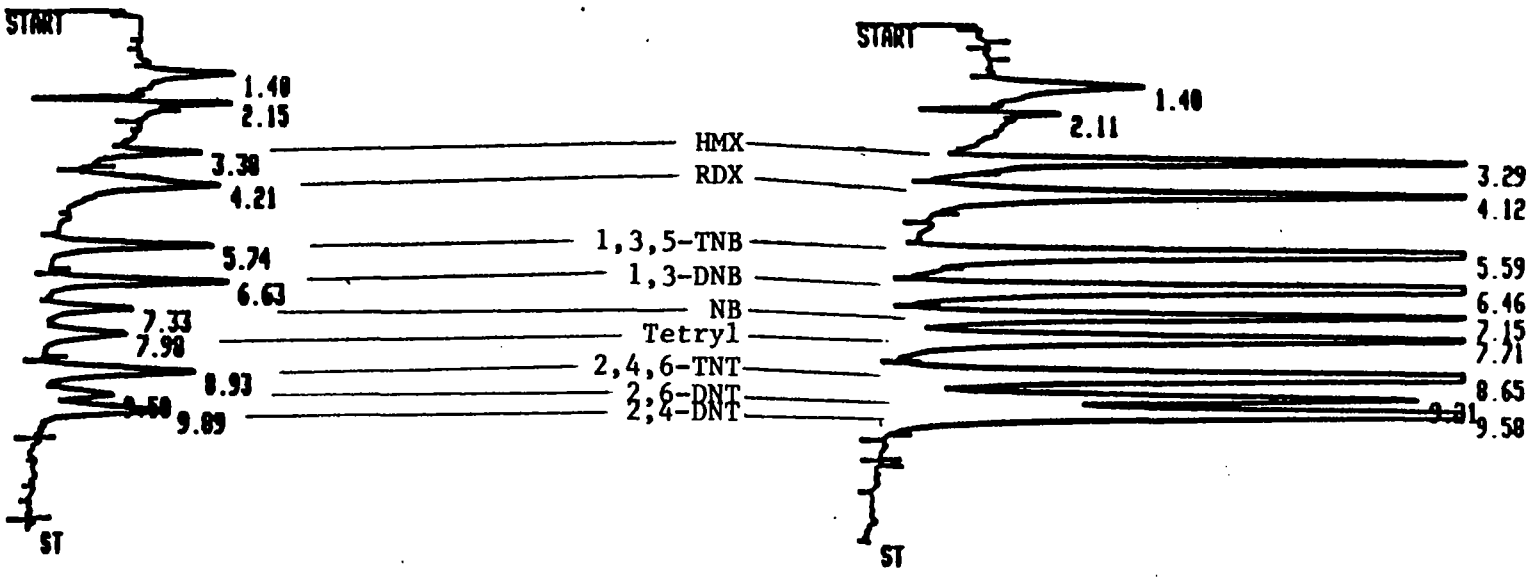
APPENDIX I: CHROMATOGRAMS

EXPLOSIVES IN SOIL - ACETONITRILE EXTRACTION

APPENDIX I: CHROMATOGRAMS
EXPLOSIVES IN SOIL - ACETONITRILE EXTRACTION

A. Near the detection Limit:
0.2X (~0.5 ug/g)

B. At approximately 10 times the detection limit: 2X (~ 5 ug/g)



RUN # 145 APR/22/83 12:58:26
ID 1

HEIGHT%

RT	HEIGHT	TYPE	AR/HT	HEIGHT%
1.40	583	VV	0.435	11.623
2.15	535	PB	0.135	10.666
3.38	332	VB	0.155	6.619
4.21	520	BV	0.365	10.367
5.74	573	PB	0.201	11.423
6.63	632	BP	0.200	12.600
7.33	305	PV	0.208	6.081
7.98	280	VB	0.258	5.582
8.93	539	BV	0.251	10.746
9.50	259	VV	0.243	5.164
9.89	458	VB	0.206	9.131

RUN # 148 APR/22/83 14:04:05
ID 1

HEIGHT%

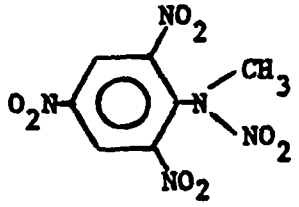
RT	HEIGHT	TYPE	AR/HT	HEIGHT%
1.40	596	BV	0.344	2.107
2.11	444	PV	0.137	1.570
3.29	2399	PB	0.158	8.483
4.12	2392	BB	0.207	8.458
5.59	4384	PB	0.186	15.502
6.46	4822	PB	0.191	17.050
7.15	2182	BV	0.193	7.715
7.71	2286	VB	0.247	8.083
8.65	3505	BV	0.241	12.393
9.21	1830	VV	0.227	6.471
9.58	3441	VB	0.251	12.167

APPENDIX II

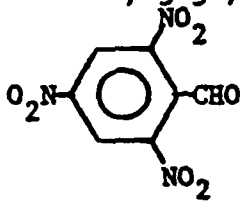
CHEMICAL FORMULAS AND STRUCTURES

APPENDIX II

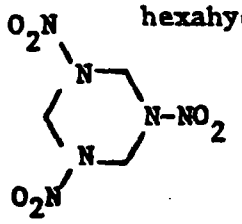
Tetryl $C_7H_5N_5O_8$ N-methyl-N,2,4,6-tetranitroaniline
2,4,6-trinitrophenylmethylnitramine



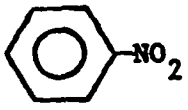
2,4,6-TNBA $C_7H_3N_3O_7$ 2,4,6-trinitrobenzaldehyde



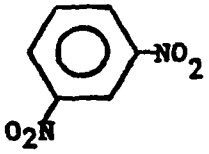
RDX $C_3H_6N_6O_6$ hexahydro-1,3,5-trinitro-1,3,5-triazine
cyclonite
hexagen
hexahydro-1,3,5-trinitro-s-triazine



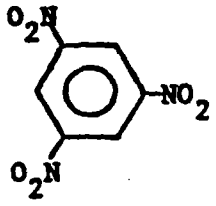
NB $C_6H_5NO_2$ nitrobenzene



1,3-DNB $C_6H_4N_2O_4$ 1,3-dinitrobenzene

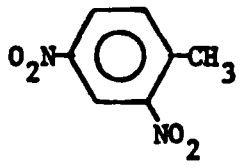


1,3,5-TNB $C_6H_3N_3O_6$ 1,3,5-trinitrobenzene

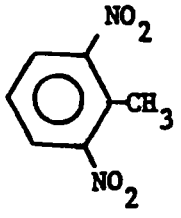


APPENDIX II
(continued)

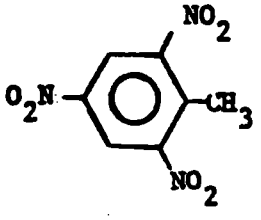
2,4-DNT $C_7H_6N_2O_4$ 2,4-dinitrotoluene



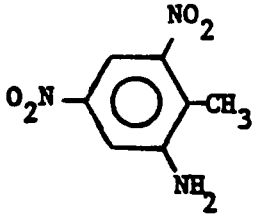
2,6-DNT $C_7H_6N_2O_4$ 2,6-dinitrotoluene



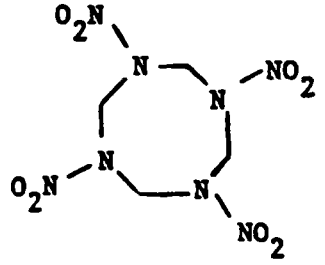
2,4,6-TNT $C_7H_5N_3O_6$ 2,4,6-trinitrotoluene
TNT



2-NH_2-4,6-DNT $C_7H_7N_3O_4$ 2-amino-4,6-dinitrotoluene
4,6-dinitro-o-toluidine



HMX $C_4H_8N_8O_8$ Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine



Work Plan Appendix B
Site Sampling Plan

Remedial Investigation/ Feasibility Study

Crab Orchard National Wildlife Refuge

U.S. Fish and Wildlife Service
U.S. Department of Interior
Marion, Illinois
and
Sangamo-Weston, Inc.
Atlanta, Georgia

June 1985

APPENDIX B

SITE SAMPLING PLAN

REMEDIAL INVESTIGATION/FEASIBILITY STUDY

CRAB ORCHARD NATIONAL WILDLIFE REFUGE

U.S. FISH AND WILDLIFE SERVICE

U.S. DEPARTMENT OF INTERIOR

MARION, ILLINOIS

AND

SANGAMO-WESTON, INC.

ATLANTA, GEORGIA

O'BRIEN & GERE ENGINEERS, INC.
1304 BUCKLEY ROAD
SYRACUSE, NEW YORK 13221

JUNE 1985

TABLE OF CONTENTS

OBJECTIVE

The objective of this Sites Sampling Plan (SSP) is to document the sampling locations, procedures and practices that will be used in the Remedial Investigation sampling program to be conducted at Crab Orchard National Wildlife.

TYPES OF SAMPLES

Various matrices will be sampled and analyzed as part of the Remedial Investigation. These include the following:

1. Waters: including groundwaters, surface streams, raw and finished water supplies, pond waters and waters from Crab Orchard Lake.
2. Sediments: from streams, ponds and Crab Orchard Lake.
3. Soils: including soils potentially affected by surface spillage and fill material from sites of past disposal activity.
4. Air: as part of the site safety program.
5. Biota: including fish, turtles and crayfish.

For the most part, all samples will be obtained as single grab samples. No time-composited samples are contemplated at this time. However, at many sites, areal soil composites will be prepared. Areal composites are used as a screening device to allow initial assessments of broad areas for a range of contaminants. Compositing procedures are discussed below.

COMPOSITING PROCEDURES

Areal composites of water samples (along stretches of streams, surfaces of ponds or depth composites in Crab Orchard Lake) will be prepared by combining equal volumes of grab samples at each location.

Individual grab samples for volatile organic analyses will be retained and labelled in individual headspace-free vials for compositing by the laboratory (see pages 25-28 and 57 of Attachment 2 to this SSP).

Areal composites of soil samples will be prepared either in the field or in the laboratory after refrigerating individual grabs to 0 to 4°C. This will minimize loss of volatile materials. Where soils are obtained in Lexan cores, these will be capped and refrigerated prior to compositing.

GENERAL SAMPLING LOCATIONS AND NUMBERS

SAMPLE LOCATIONS

Sampling locations were determined in the field during a site reconnaissance visit on March 26-28, 1985. They are presented in Attachment 1 of this SSP. A log book listing the various samples to be collected will be prepared for use on-site. The log book will also contain the type of sample and analytical matrix for each of the samples to be collected. Pre-printed peel-off labels will be included in the log book for tagging the various containers to be used for sample collection. The sample team leader will be responsible for determining the exact sampling location and recording the location in the field sampling notebook. The location will be described in the log book with a sketch that includes distances from numbered field reconnaissance stakes and other landmarks. The rationale of selecting a sampling location will also be included. All sampling locations will be photographed.

SAMPLE NUMBERING SYSTEM

A sample numbering system will be used to identify each sample taken during the remedial investigation sampling program. This numbering system will provide a tracking procedure to allow retrieval of information regarding a particular sample and to assure that each sample is uniquely numbered. A listing of the sample identification numbers will be maintained by the sample team leader.

SAMPLING EQUIPMENT AND SAMPLING PROCEDURES

SOIL SAMPLING

Soil samples will be collected from identified spots around the Refuge and during the installation of additional groundwater monitoring wells. Samples will be collected in general accordance with the split spoon sampling procedure (ASTM D1586-67), using 2-inch OD split spoon samplers. See also the protocols described in the Addendum to Attachment 1 of this SSP.

GROUNDWATER STUDIES AND SAMPLING

Aquifer slug recovery tests will be conducted in all additional monitoring wells to obtain in situ estimates of hydraulic conductivity. A minimum of two test runs should be made at each test well.

Properly decontaminated equipment will be used in sampling all groundwater monitoring wells. See the Decontamination protocols in Attachment 3 of the QAPP. Before samples are taken, each well will be purged until there is a constant conductivity, (usually about 5 to 10 well volumes). After the well has recovered, samples for inorganic and organic (excluding volatiles) analysis can be collected using a peristaltic

pump or hand bailer. Samples to be analyzed for volatile organics will be collected by bailing. See also the further discussion in Attachment 1 of this SSP.

Teflon tubing will be used for the suction and discharge lines for peristaltic pumps. Hand bailers will be constructed of stainless steel or Teflon.

WASTE SAMPLING

The Area 9 Landfill is the only site of the Refuge where waste materials are being sampled. All other sites represent sampling of matrices potentially affected by dispensed contaminants. There are special safety concerns posed by the sampling of waste materials at Area 9 because of the possible presence of explosives residues or even undetonated cartridges. Similar concerns exist at other sampling sites, but sampling elsewhere is limited to within 1 foot from the surface. Soil borings at Area 9 will employ split spoon sampling procedures. Drilling personnel will be required to be removed at least 100 ft. from the drill rig during advancement of the augers. This is further discussed in the SHSP.

FIELD BLANKS

Field Blanks for sediment and soil samples will consist of analytical grade diatomaceous earth. For water samples, ultrapure distilled/deionized water will be used. The field blank sample will be placed into the appropriate sampling equipment, removed from the equipment, and then placed into sampling containers.

DUPLICATE SAMPLES

Duplicate samples are defined as two distinct samples taken from the same location at similar times using identical sampling equipment that has been decontaminated in a similar manner. However, duplicate samples of soil cores will consist of a given core homogenized, divided equally and submitted for analysis as two distinct samples.

SPLIT SAMPLES

A number of samples will be split with a representative of the FWS for analysis. Split samples are defined as one distinct sample that is divided equally and sent to two different laboratories for analysis. Soils will be field homogenized in a clean aluminum pan prior to splitting. Water sample splits will be duplicates.

GENERAL DECONTAMINATION PROCEDURES

Decontamination of personal gear (boots, gloves, and waders), sample jars and sampling equipment will be as follows (see also attached materials to the SHSP):

1. Wash personal gear or sample containers in a bucket or tub filled between 50 and 75 percent with a trisodium phosphate (TSP) solution (2 lbs of TSP per 10 gallons of clean water). Completely brush the entire exterior surface of the article undergoing decontamination. If PCB's are expected to be present, add 4 lbs of sodium bicarbonate per 10 gallons of water to the washing solution.
2. Rinse personal gear or sample containers in a bucket or tub filled between 50 and 75 percent with clean water. Completely brush the entire exterior surface of the article undergoing decontamination.

3. Dispose of all wash and rinse water in a properly marked and sealed container. All such containers of wastewater will be stored in a secure area on-site and properly disposed of during the remedial action phase.

SAMPLING EQUIPMENT

1. Wash sampling equipment in a bucket or tub filled between 50 and 75 percent with a TSP solution (2 lbs of TSP per 10 gallons of clean water). Completely brush the entire exterior surface of the article undergoing decontamination. Wash interior wetted surfaces as required. If PCB's are expected to be present, add 4 lbs of sodium bicarbonate to the washing solution. Drilling equipment, augers and split spoon samplers can be decontaminated by steam cleaning using clean water.
2. Rinse only heavily contaminated sampling equipment in a bucket or tub filled between 50 and 75 percent with a 20 percent solution of acetone and water. Completely brush the entire exterior surface of the article undergoing decontamination. Rinse interior wetted surfaces as required. If PCB's are present, the first rinse should be carried out with a hexane solution.
3. Following step 2 above, rinse all sampling equipment in a bucket or tub filled between 50 and 75 percent with distilled water. Completely brush the entire exterior surface of the article undergoing decontamination. Rinse interior wetted surfaces as required.

4. Collect all wash and rinse water in a properly marked and sealed container. Wash and rinse water will be analyzed relative to its hazardous waste characteristics and disposed of in accordance with all applicable state and federal regulations. Drilling soils and water as well as discarded protective clothing will be treated similarly.

SCREENING PROCEDURES

It is probable that not all soil samples will have significant concentrations of contaminants. To reduce analytical costs, a field screening procedure may be used in Phase II of the RI to reduce the number of soil samples sent for complete laboratory analysis.

While constituents used for screening may not be the only contaminants present, they may be used as an indicator of contamination. If they are present in a sample in concentrations exceeding the positive response criteria established in the Work Plan, the interpretation that other contaminants may also be present will be made and the sample will be sent to the laboratory for analysis of constituents established in the Work Plan.

DOCUMENTATION

SITE LOCATION PROCEDURE

Following sampling location identification, a wood stake (approximately 2" X 2" X 24") will be driven into the ground, allowing approximately 8 to 10 inches of the stake to remain visible above ground. The top portion of the stake will be painted orange and

labeled for identification. The label will contain sample number and sample type. The location of each stake will be recorded. Sample locations will eventually be surveyed and tied into the site grid system.

PHOTOGRAPHS

Photographs (35mm, color slides) will be taken to illustrate sampling locations. Photographs will show the surrounding area and reference objects which help to locate sampling sites. The picture number and roll number (if more than one roll of film is used) will be logged in the field notebook to identify which sampling site is depicted in the photograph. The film roll number will be identified by taking a photograph of an informational sign on the first frame of the roll. This sign would have the job and film roll number written on it to identify the pictures contained on the roll.

FIELD NOTEBOOKS

Field notebooks will provide the means of recording data on collecting activities performed at a site. As such, entries will be described in as much detail as possible so that anyone going to the site could reconstruct a particular situation without reliance on memory.

Field notebooks will be bound. Notebooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each notebook will be identified by the project-specific document number.

The cover of each notebook will contain:

Person or Organization to whom the book is assigned.

Book Number

Project Name

Start Date

End Date

Entries into the notebook will contain a variety of information. At the beginning of each entry, the date, start time, weather, all field personnel present, level of personal protection being used onsite, and the signature of the person making the entry will be entered. The names of visitors to the site, all field sampling team personnel and the purpose of their visit will be recorded in the field notebook.

All measurements made and samples collected will be recorded. All entries will be made in ink with no erasures allowed. If an incorrect entry is made, it will be crossed out with a single strike mark. Wherever a sample is collected or a measurement is made, a detailed description of the location of the station, which includes compass and distance measurements, shall be recorded. The film roll number and number of photographs taken of the station will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the procedures documented in this plan. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. In addition, the cooler number into which the sample is placed in the field will be recorded. Sample numbers will be assigned prior to going onsite.

Duplicates, which will receive an entirely separate sample number, will be noted under sample description. Significant field notebook entries (samples collected, significant observations) shall be countersigned by another member of the project team.

CONTROL OF CONTAMINATED SAMPLING MATERIALS

Disposable sampling and safety equipment and excess samples may be generated during sampling operations. These materials will be placed in 55-gallon drums (separate drums for solids, decontamination liquids, debris, and disposable equipment). Decontamination liquids should also be separated based on those containing solvents (acetone, hexane, etc.) and those containing only detergents (TSP, etc.). The drums will be sealed, labelled and properly stored in a secure area for proper, legal disposal during the remedial action phase. Bailed well water and contaminated drilling spoils will be drummed for proper storage in a secure area.

SAMPLE CONTROL

Serialized sample tags will be used to label each sample for analysis. Chain-of-custody records will be completed for all samples according to EPA requirements and procedures set forth in NEIC Policies and Procedures EPA-330-19-78-001R. Custody seals will be placed on all shipping coolers containing samples.

SAMPLE CONTAINERS AND SAMPLE PRESERVATION

Required sample containers, filling instructions and preservation procedures are listed in Table 1 of Attachment 1 of this SSP. The

collected samples will be kept out of direct sunlight and, after decontamination and labeling, will be placed in coolers for shipment to the analytical laboratory.

SAMPLE SHIPPING

Samples will be packed and labelled according to DOT regulations and protocols appearing in Attachment 1 of this SSP. Samples will be shipped via a 24 hour delivery service to the analytical laboratory so that the samples can be extracted within allowable time limits (See QAPP).

ATTACHMENT 1

SITE INVESTIGATION PROGRAMS

CONTENTS

22. Old Refuge Shop

24. Pepsi-West

25. COC at Marion Landfill

26. COC bellow Marion STP

27. COC below 157 Dredge Area

28. Water Tower Landfill

29. Fire Station Landfill

30. Munition Control Site

31. Refuge Control Site

32. Area 9 Landfill

33. Area 9 Building Complex

34. Crab Orchard Lake

INTRODUCTION

This Attachment presents the site background and Phase I sampling schedules for each site at Crab Orchard National Wildlife Refuge included in the RI/FS. The general site investigation rationale is discussed under Task 3 of the Work Plan.

The site background discussions represent information provided by the Refuge Manager and also include observations developed as a result of site visits conducted during the period of March 26 through 28, 1985.

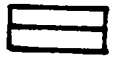
In general, the enclosed sampling and analysis schedules represent Phase I activities only. Based on the results of these analyses, a Phase II sampling and analysis program will be developed to more fully assess the extent of contamination (lateral and vertical) and the involvement with site receptors (groundwater, soils, surface waters, air, aquatic biota, etc.).

The proposed sampling locations are illustrated on aerial photo overlays for each of the sites. The date of the aerial photo is indicated on each. Composite samples are illustrated by the dotted lines connecting the compositing locations. The sampling and analysis schedules further define the depth of samples and number of grabs within each composite. Groundwater monitor wells and geophysical survey grid lines are also illustrated, where proposed.

Key

Sample compositing location: ●--●--●

Groundwater Monitor Well: 

Geophysical Survey Grid: 

A listing of the parameters to be analyzed in the various samples collected around the Refuge is given in Table 1 of this attachment. The analysis sets included in the sampling and analysis schedule are also shown in Table 1. In Analysis Sets A, C & D, the screening procedures described in Appendix A (QAPP) for priority pollutants and PCDD/PCDF will be followed.

Full priority pollutants and PCDD/PCDF will be analyzed in Analysis Sets F, G & H. A summary of the sampling and analysis schedule for each site is presented as Table 2.

TABLE 1

Parameters	Analysis Set A	B	C	D	E	F	G	H
1. Purgeable Priority Pollutants (Screening and Full Analysis)	x	-	-	x	-	x	x	-
2. Acid Extractable Priority Pollutants (Screening and Full Analysis)	x	-	-	x	-	x	x	-
3. Base/Neutral Extractable Priority Pollutants (Screening and Full Analysis)	x	-	-	x	-	x	x	-
4. Pesticide/PCB Priority Pollutants (Screening and Full Analysis)	x	-	-	x	-	x	x	-
5. PCB's	-	x	x	-	-	-	-	-
6. Metals								
- ICP scan	x	-	-	x	-	-	-	-
- Priority Pollutant Metals by AA Spec	-	-	-	-	-	x	x	-
- Mercury	x	-	-	x	-	-	-	-
7. Cyanide 40	x	-	-	x	-	-	-	-
8. Indicators								
- pH (field)	x	-	x	x	-	-	-	-
- Secific Conductance (field)	x	-	x	x	-	-	-	-
- Total Organic Carbon	x	-	x	x	-	-	-	-
- Total Organic Halogens	x	-	x	x	-	-	-	-
9. Explosives Residues by HPLC	x	-	-	x	-	-	-	-
10. Nitrogen Series: TNK, NH3, NO3	x	-	x	x	-	-	x	-
11. PCDD/PCDF (Screening and Full Analysis)	-	-	x	x	-	-	x	x
12. Cation Exchange Capacity	-	-	x	-	-	-	-	-
13. Total Phosphorus	x	-	-	x	-	-	-	-
14. Primary and Secondary Drinking Water Standards	-	-	-	-	x	-	-	-

Note: Procedures for Screening and Full Analysis are referenced in the Work Plan.

Analysis sets F, G, and H are full analysis of selected samples instead of screening for parameters as noted in Sets A, D and C respectively.

TABLE 2

RI/FS SAMPLING & ANALYSIS SUMMARY

SITE NO.	SAMPLE TYPE	WATER NO. OF SAMPL	WATER ANAL. TYPE	WELL NO. OF SAMPL	WELL ANAL. TYPE	SOILS NO. OF SAMPL	SOILS ANAL. TYPE	SEDIMENTS NO. OF SAMPL	SEDIMENTS ANAL. TYPE	BIOTA NO. OF SAMPL	BIOTA ANAL. TYPE
3	AREA 11 SOUTH LANDFILL	0	-	0	-	3	A	1	A	0	-
						1	F	1	D		
4	AREA 11 NORTH LANDFILL	0	-	0	-	1	D	1	A	0	-
5	AREA 11 ACID POND	1	A	0	-	1	A	1	A	0	-
								1	F		
7 D	AREA SOUTHEAST DRAINAGE	1	A	0	-	0	-	1	A	0	-
8 D	AREA SOUTHWEST DRAINAGE	1	A	0	-	0	-	1	A	0	-
9 D	AREA NORTHWEST DRAINAGE	1	A	0	-	0	-	1	A	0	-
10	WATERWORKS NORTH DRAINAGE	1	A	0	-	0	-	1	D	0	-
								1	G		
11 P	AREA SOUTHEAST DRAINAGE	1	A	0	-	0	-	1	A	0	-
7A D	AREA NORTH LAWN	0	-	0	-	16	A	0	-	0	-
						2	F				
11A P	AREA NORTH	0	-	0	-	4	A	4	A	0	-
12	AREA 14 LANDFILL	1	A	0	-	1	A	1	D	0	-
								1	G		
13	AREA 14 CHANGE HOUSE SITE	0	-	0	-	6	A	0	-	0	-
						1	F				
14	AREA 14 SOLVENT STORAGE	2	A	0	-	0	-	2	A	0	-
15	AREA 7 PLATING POND	1	A	1	A	0	-	1	A	0	-
16	AREA 7 INDUSTRIAL SITE	4	A	0	-	7	A	4	A	0	-
		1	F			2	D	1	F		
						1	F				
						1	G				
17	JOB CORPS LANDFILL	2	A	4	A	5	A	0	-	0	-
		1	F	1	F	2	D				
						1	F				
						1	G				
18	AREA 13 LOADING PLATFORM	0	-	0	-	4	A	0	-	0	-
						1	F				
19	AREA 13 BUNKER 1-3	0	-	0	-	5	A	0	-	0	-
						1	F				

Table 2 (Contd)

RI/FS SAMPLING & ANALYSIS SUMMARY

SITE NO.	SAMPLE TYPE	WATER NO. OF SAMPL	WATER ANAL. TYPE	WELL NO. OF SAMPL	WELL ANAL. TYPE	SOILS NO. OF SAMPL	SOILS ANAL. TYPE	SEDIMENTS NO. OF SAMPL	SEDIMENTS ANAL. TYPE	BIOTA NO. OF SAMPL	BIOTA ANAL. TYPE
30	MUNITIONS CONTROL SITE	0	-	1	A	1	D	0	-	0	-
20	D AREA SOUTH	1	A	0	-	0	-	1	A	0	-
21	SOUTHEAST CORNER FIELD	0	-	0	-	4	A	0	-	0	-
						1	F				
22	OLD REFUGE SHOP	1	A	0	-	0	-	1	A	0	-
24	PEPSI-WEST	1	A	0	-	0	-	1	A	0	-
25	C.O.CREEK AT MARION LF	3	A	0	-	0	-	2	A	0	-
								1	D		
								1	G		
26	C.O.CREEK BELOW MARION STP	2	A	0	-	0	-	2	A	0	-
27	C.O.CREEK BELOW 157 DREDGE	1	A	0	-	0	-	1	D	0	-
28	WATER TOWER LANDFILL	0	-	2	A	11	A	0	-	0	-
				1	F	1	D				
						3	F				
29	FIRE STATION LANDFILL	0	-	4	A	5	A	0	-	0	-
				3	F	2	D				
						2	F				
31	REFUGE CONTROL SITE	0	-	1	A	1	D	0	-	0	-
32	AREA 9 LANDFILL	0	-	3	A	0	A	15	A	0	-
				3	F	6	B	3	D		
						27	C	3	F		
						9	D	1	G		
						3	G				
						6	H				
33	AREA 9 BUILDING COMPLEX	0	-	0	-	201	B	0	-	0	-
34	CRAB ORCHARD LAKE	10	A	0	-	0	-	5	A	21	A
		5	E					1	F	5	F
	TOTAL NUMBER OF ANALYSES	42		24		350		63		26	
	505										

NOTE: ANALYSIS SETS F, G & H are full analysis of selected samples collected for SETS A,B,C,D & E

Table 2 (Contd)

RI/FS SAMPLING & ANALYSIS SUMMARY

PHASE I SAMPLING AND ANALYSIS SUMMARY
ANALYSIS SET

NO. OF ANALYSES	SCREENING A	B	C	D	E	TOTAL NO. OF SAMPLES	FULL ANALYSIS F	G	H	TOTAL ANALYSIS
WATER	35	0	0	0	5	40	2	0	0	42
WELL	16	0	0	0	0	16	8	0	0	24
SOILS	72	207	27	19	0	325	14	5	6	350
SEDIMENTS	45	0	0	8	0	53	6	4	0	63
BIOTA	21	0	0	0	0	21	5	0	0	26
TOTAL NO.	189	207	27	27	5	455	35	9	6	505
QA/QC Samples	28	31	4	4	1	68	5	2	1	76

PHASE II SAMPLING AND ANALYSIS CONTINGENCY

NO. OF SAMPLES	EXPLOSIVES	METALS	PCBs	ANALYSIS PARAMETERS PCDDs/ PCDFs	PRIORITY POLLUTANT ORGANICS	MISC.	TOTAL
WATER	10	10	0	0	10	10	40
WELL	10	10	0	10	10	10	50
SOILS	41	41	0	0	10	41	133
SEDIMENTS	14	14	14	0	0	14	56
BIOTA	0	0	0	0	0	0	0
TOTAL NO.	75	75	14	10	30	75	279
QA/QC Samples	28	31	4	4	1	5	73

SITE 3

AREA 11 SOUTH LANDFILL

Background

Areas 11 and 12 are currently abandoned sites of explosives and nitrogen fertilizer manufacturing as well as munitions loading. The Olin Corporation is reported to have operated a dynamite line there which was later reportedly sold to U.S. Powder. A number of fires and explosions are known to have occurred in these areas. Use of lead azide in the area is suspected. RDX may have been used in this area. Many of the buildings and grounds have been "torched" to remove residuals of flammable material. Most of the buildings are covered with a spark-retarding asbestos siding material. Also, within Area 11 are storage areas where explosive powders were stored in rubber-lined underground trenches. A burning pad is evident to the south of Area 11 where oil residues, 53-calibre powder magazines and small powder cylinders are noticeable on the surface. The evaluations of these areas are not included in this scope of work.

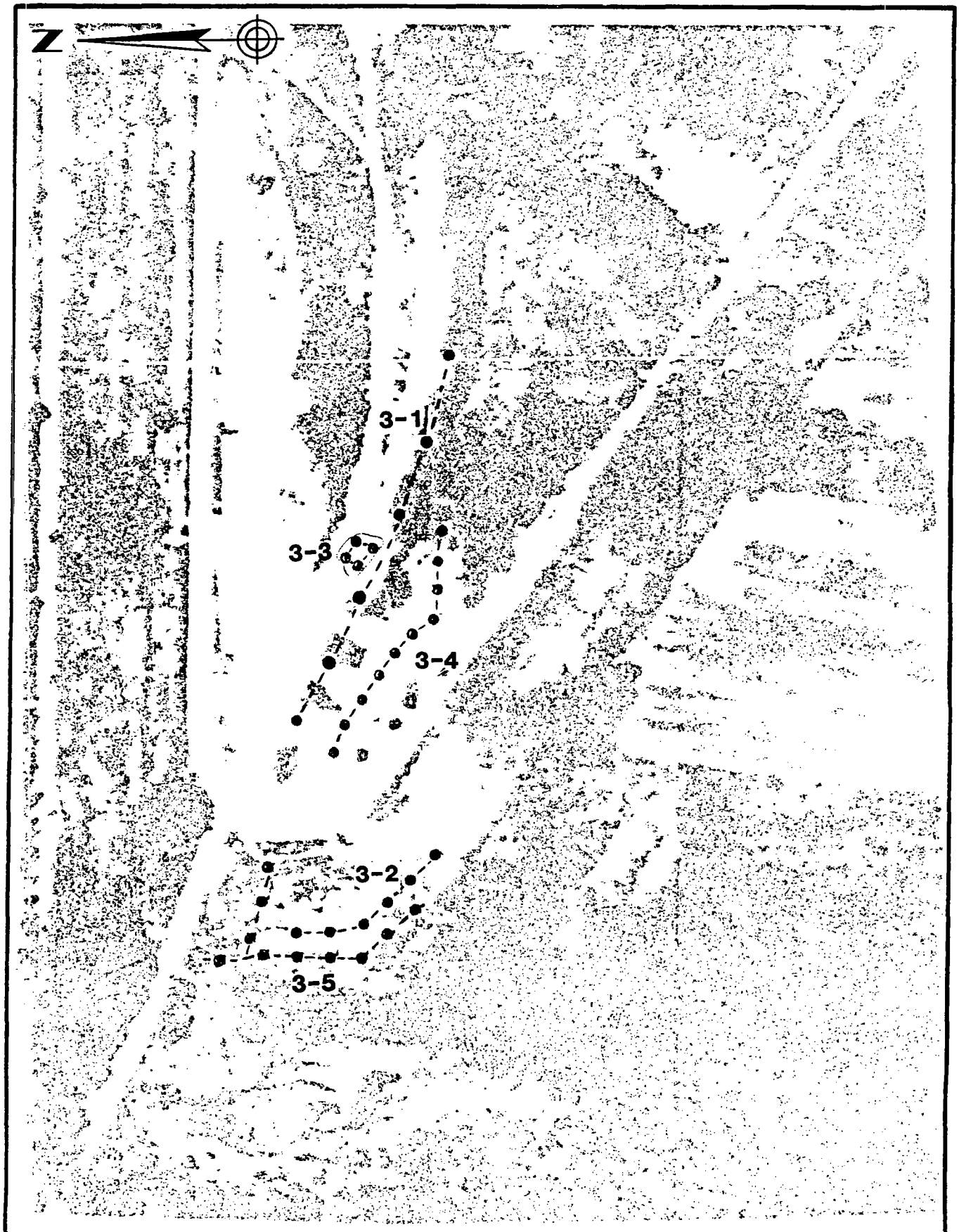
The Area 11 South Landfill is located adjacent to what appears to be an old railroad bed. Much surface and buried litter is evident over an area of perhaps 10 acres. In addition to railroad track, ties and ballast, the following were also observed: cinders and charred wood, powder canisters, piping, metal, mesh, bricks, pumice blocks, 30- and 55-gal drums, reinforcing bars, a laboratory flask and miscellaneous wire and plastic articles. One mound on the bank just above the stream bed has several of what appeared to be metal vents on the top and a 4-in stainless steel pipe drain extending from the bottom. The

stream bed west of the road appeared to contain especially heavy concentrations of debris. Black tars and ash were evident in the stream bed.

Sampling and Analysis Schedule

Phase I sampling only will be conducted at this site. Any Phase II sampling and remedial assessments will likely be conducted by the DOD. The following samples are proposed:

I.D.	Matrix	Name	Type	Depth	Analysis Set
3-1	Soil	North Bank	Composite of 6 Grabs	0 to 1 ft	A
3-2	Soil	South Bank	Composite of 6 Grabs	0 to 1 ft	A
3-3	Soil	East Mound	Composite of 4 Grabs	0 to 1 ft	A
3-4	Sed.	Marsh	Composite of 10 Grabs	0 to 1 ft	D
3-5	Sed.	Lower Stream	Composite of 10 Grabs	0 to 1 ft	A



SITE 3

Area 11 South Landfill

100 ft (approx)

O'BRIEN & GERE ENGINEERS, INC

SITE 4

AREA 11 NORTH LANDFILL

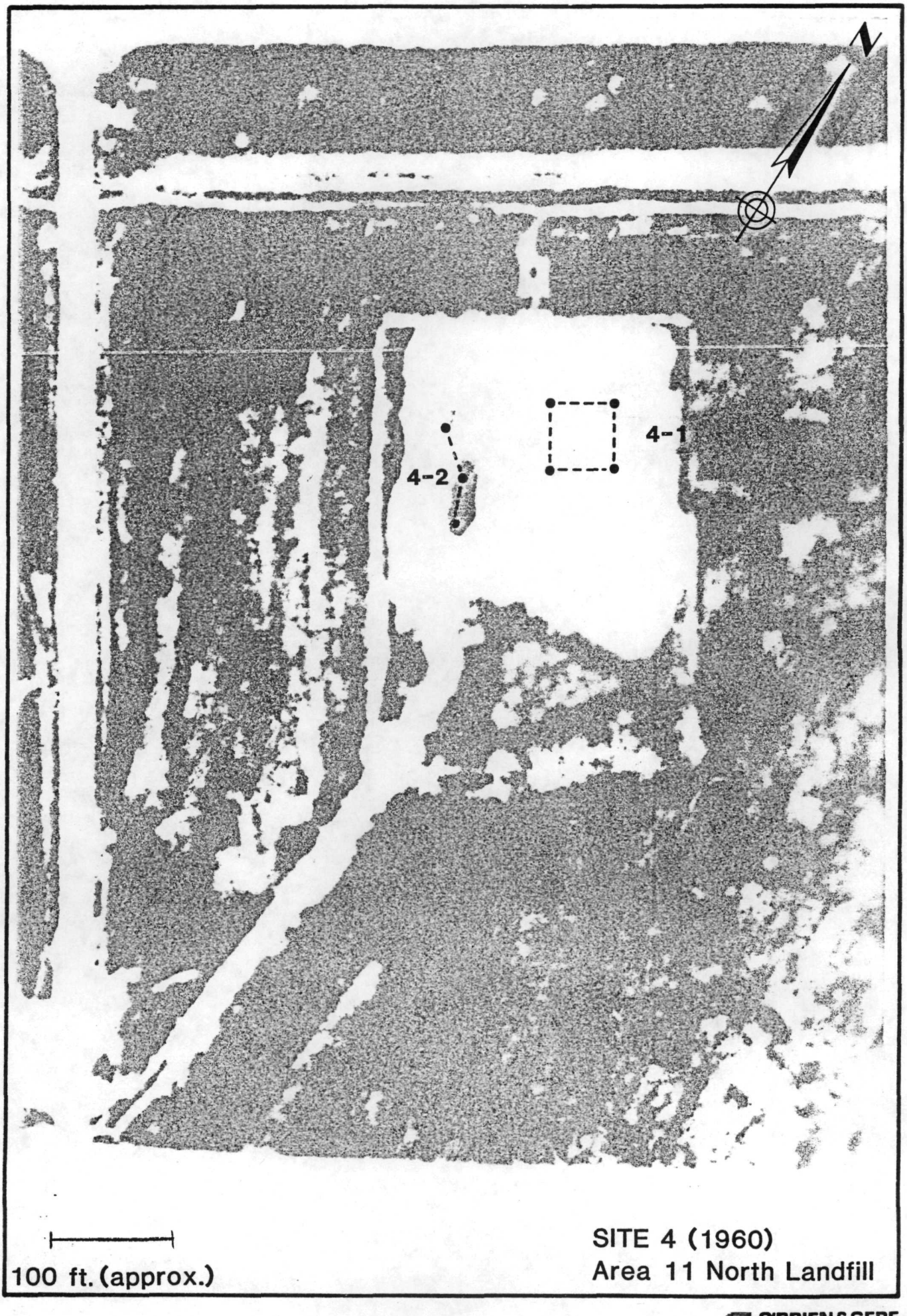
Background

The Area 11 North Landfill appears to have been the site of a large (2 to 3 acre) impoundment. The impoundment is flat in the middle and has small intermittent stream or marsh areas bordering the east and west boundaries. Water appears to flow from south to north following periods of precipitation. The reinforced concrete remains of a dam can be seen at the northwest end of the site. A large earth bunker is located immediately to the west. It may have been built with earth excavated from the semi-marshy lagoon area and may have been constructed to protect the explosives processing areas located further to the west. It was suggested that RDX or magnesium may have been stored underwater here or the area may have been used to detonate explosives or for experimental detonations. The level bottom of the impoundment shows a number of bare patches of fine white silt or clay. Other weathered areas showed horizontal layering of white and gray sediments. A number of dynamite-type fuses were noticed here as well as a small powder carrier, 1.5-in dia by 3 in, with the fuse intact. Small lead chunks were also observed.

Sampling and Analysis Schedule

Phase I sampling only will be conducted at this site. Any Phase II sampling and remedial assessments will likely be conducted by the DOD. The following samples are proposed:

I.D.	Matrix	Name	Type	Depth	Analysis Set
4-1	Soil	Bare Patches	Composite of 6 Grabs	0 to 1 ft	D
4-2	Sed.	Swampy Sed.	Composite of 6 Grabs	0 to 1 ft	A



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SITE 5

AREA 11 ACID POND

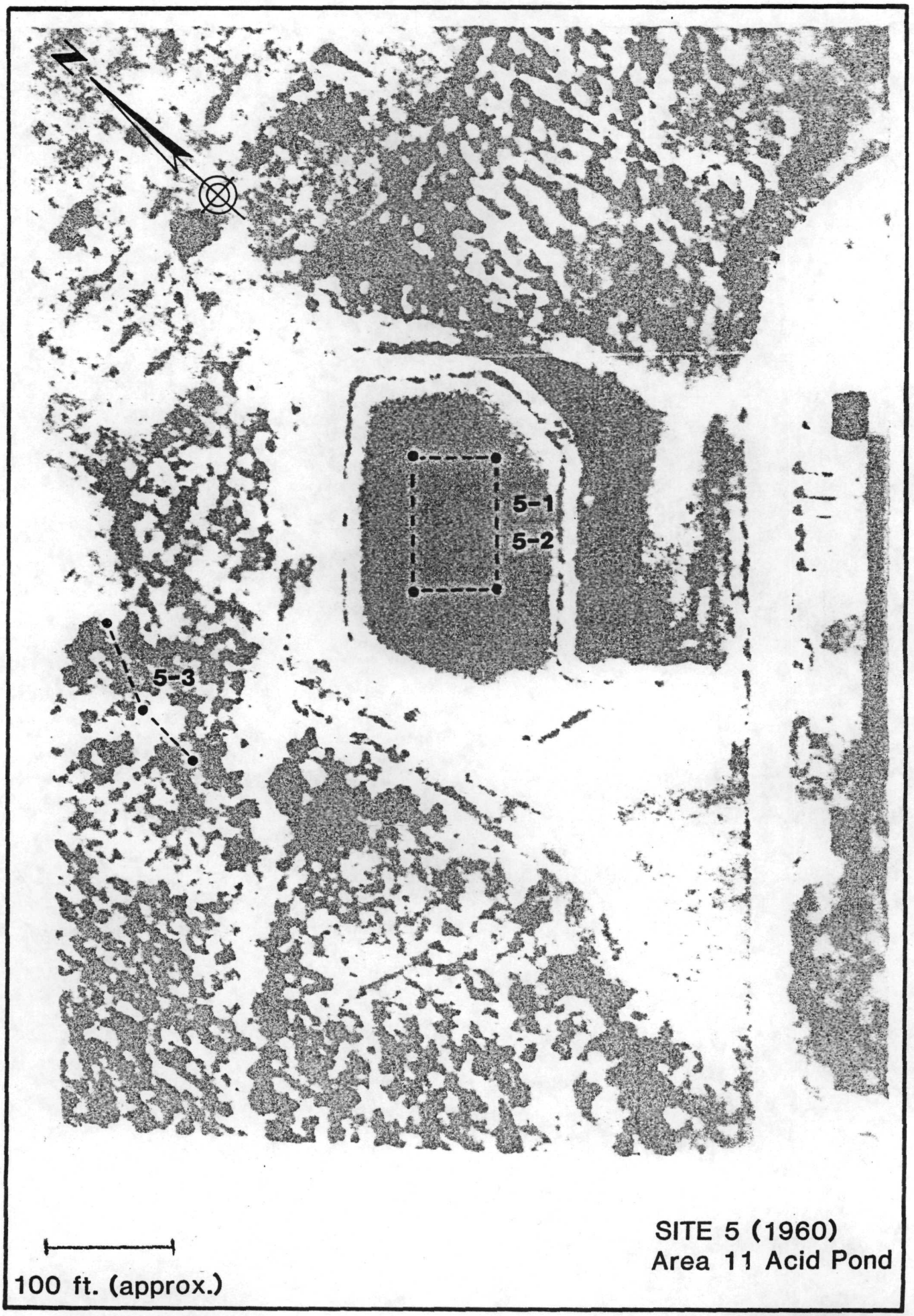
Background

The Area 11 Acid Pond is a diked impoundment approximately 300 ft x 150 ft which received drainage flowing north from the Area 11 process buildings. The dike extends 5 to 6 ft above the current water level. A 12 inch diameter pipe exits to the west through the levee to a valve box which controls the discharge from the pond to a small stream. This drainage then exits through the woods and swampy areas to the north. It is claimed that a spill of low-pH water (nitric acid) from the pond years ago killed all of the downstream vegetation for 1/4 mile. A large stand of dead trees is still visible along the creek north of the pond.

Sampling and Analysis Schedule

The following Phase I sampling effort is proposed:

I.D.	Matrix	Name	Type	Depth	Analysis Set
5-1	Water	Pond Water	Composite of 4 Grabs	Surface	A
5-2	Sed.	Pond Sed.	Composite of 4 Grabs	0 to 1 ft	A
5-3	Soil	Dead Tree Area	Composite of 4 Grabs	0 to 1 ft	A



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SITES 7, 8, 9, 10 and 11
D AREA SOUTHEAST DRAINAGE
D AREA SOUTHWEST DRAINAGE
P AREA NORTHWEST DRAINAGE
WATERWORKS NORTH DRAINAGE
P AREA SOUTHEAST DRAINAGE

Background

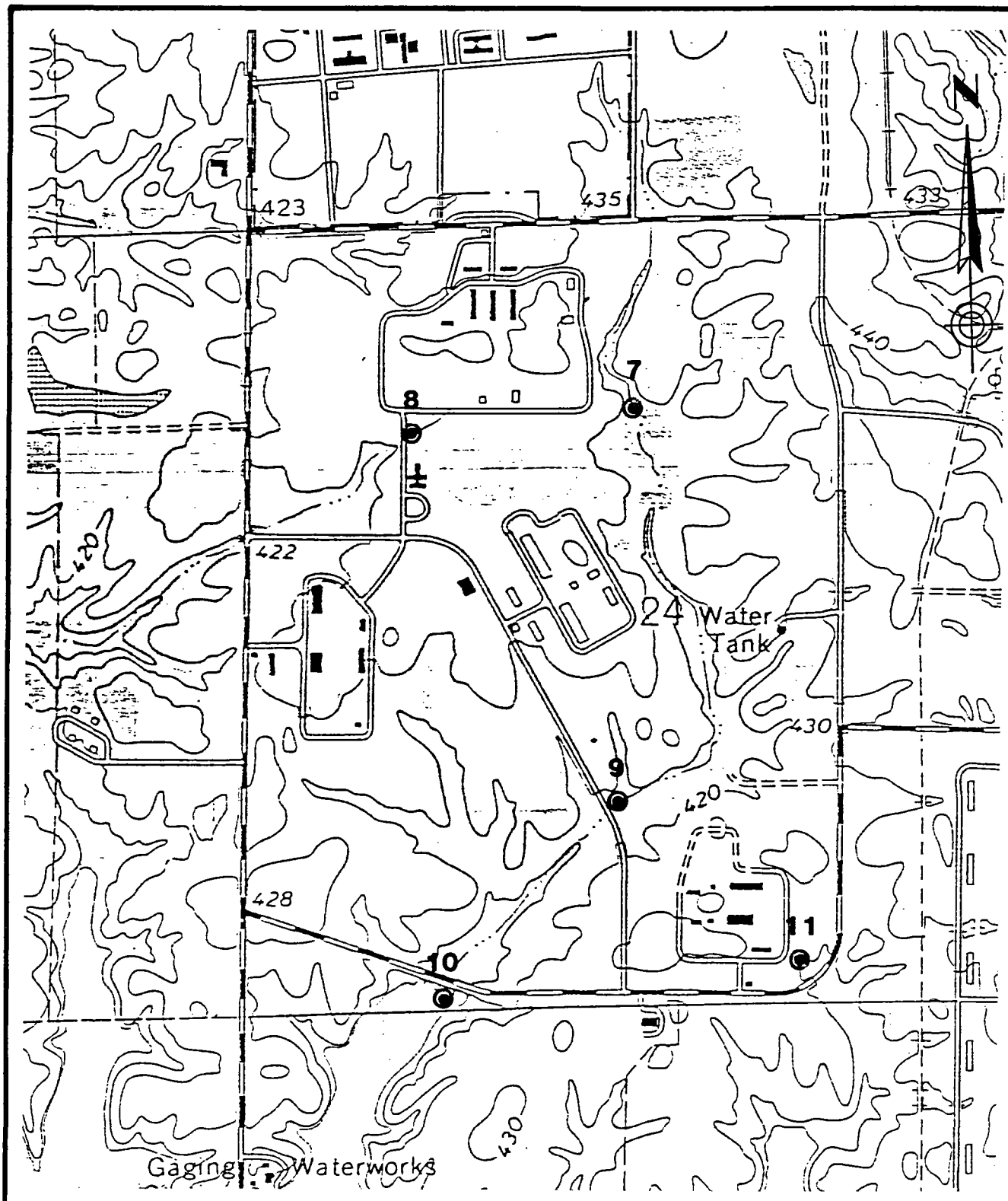
The Olin D and P Areas are active Olin operations north of Crab Orchard Lake. Explosives are currently manufactured in the D Area while research and development is conducted in the P Area. It is likely that chemicals handled in the P Area are non-conventional or "exotic". Universal Match also previously conducted operations here under contract to the DOD. Their operations ceased after a large explosion.

Sites 7, 8, 9, 10 and 11 are locations within various drainage channels leading from the Olin D and P Areas. These discharge to the Lake near the Refuge Waterworks.

Sampling and Analysis Schedule

Samples from each of these sites will consist of a water composite and sediment composite to be taken at each site as follows:

I.D.	Matrix	Name	Type	Depth	Analysis Set
7-1	Water	D-SE Water	Composite of 4 Grabs	Surf	A
7-2	Sed.	D-SE Sed.	Composite of 4 Grabs	0 to 1 ft	A
8-1	Water	D-SW Water	Composite of 4 Grabs	Surf	A
8-2	Sed.	D-SW Sed.	Composite of 4 Grabs	0 to 1 ft	A
9-1	Water	P-NW Water	Composite of 4 Grabs	Surf	A
9-2	Sed.	P-NW Sed.	Composite of 4 Grabs	0 to 1 ft	A
10-1	Water	WW-N Water	Composite of 4 Grabs	Surf	A
10-2	Sed.	WW-N Sed.	Composite of 4 Grabs	0 to 1 ft	D
11-1	Water	P-SE Water	Composite of 4 Grabs	Surf	A
11-2	Sed.	P-SE Sed.	Composite of 4 Grabs	0 to 1 ft	A



SITE 7 D Area SE Drainage
SITE 8 D Area SW Drainage
SITE 9 P Area NW Drainage
SITE 10 Waterworks North Drainage
SITE 11 P Area SE Drainage

Scale: 1" – 1000'

O'BRIEN & GERE ENGINEERS, INC.

SITE 7A

D AREA NORTH LAWN

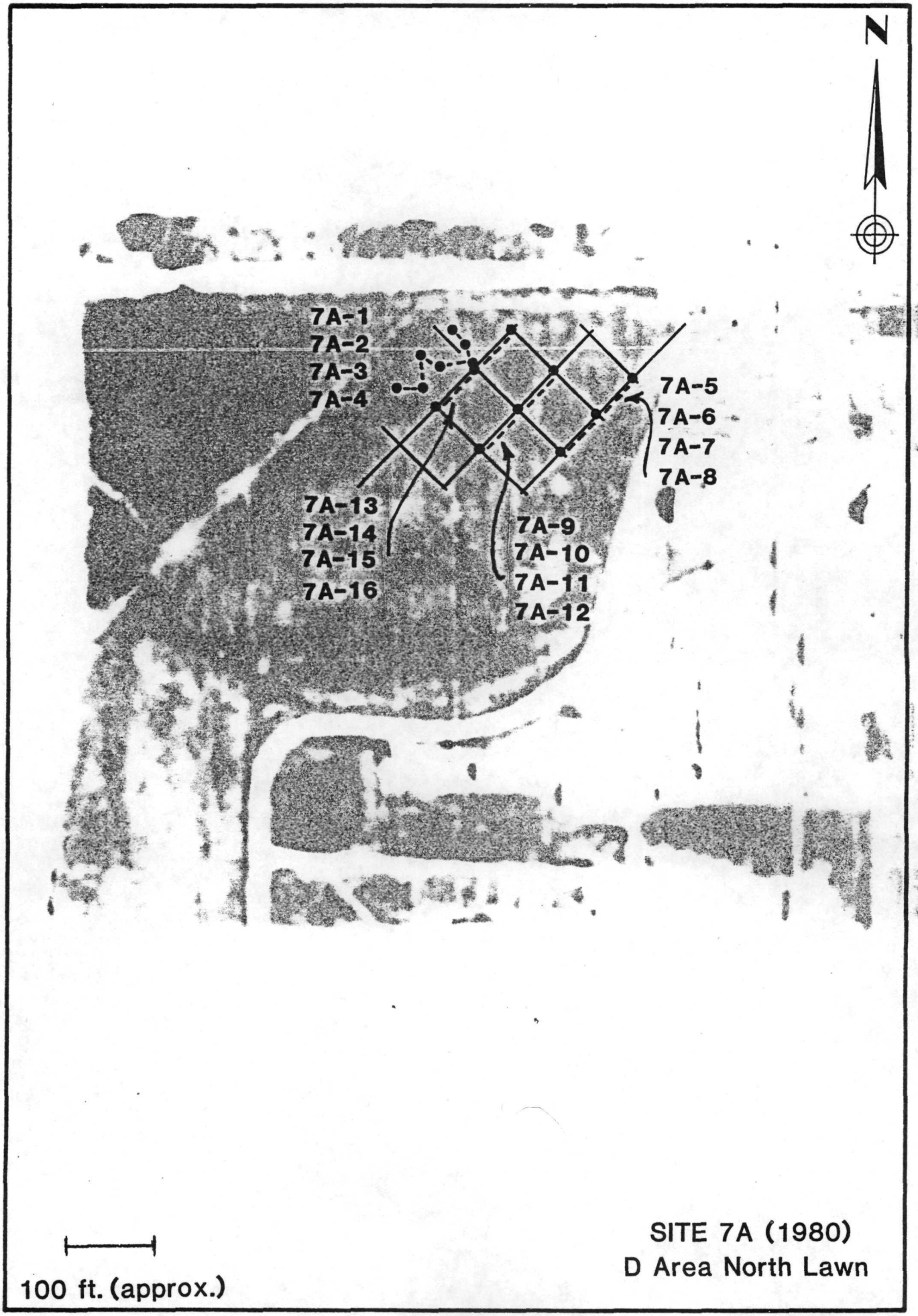
Background

There is a large (about 3 acre) lawn located northwest of the active Olin D Area complex. It is claimed that barrels of chemicals were dumped on a knoll within this lawn. No evidence of a knoll was seen during the site visit, but a number (about 8) of depressed brown patches were evident on the lawn. A visually clean drainage channel is located south of the lawn and exits under the fence to the west. Other moist drainage areas extend to the wooded area to the west of the site.

Sampling and Analysis Schedule

A magnetometer and electromagnetic (EM) survey will be conducted over the 300 ft x 200 ft lawn area on 20 ft x 20 ft grid spacings. The purpose of these surveys is to determine if any buried materials are present within the lawn area. Subsequent to the geophysical surveys, soil samples will be obtained in accordance with the following schedule:

I.D.	Matrix	Name	Type	Depth	Analysis Set
7A-1	Soil	Low spots-surf	Composite of 7 Grabs	Surf	A + OVA screen
7A-2	Soil	Low spots-1 ft	Composite of 7 Grabs	1 ft	A + OVA screen
7A-3	Soil	Low spots-2 ft	Composite of 7 Grabs	2 ft	A + OVA screen
7A-4	Soil	Low spots-3 ft	Composite of 7 Grabs	3 ft	A + OVA screen
7A-5	Soil	Transect A-surf	Composite of 3 Grabs	Surf	A + OVA screen
7A-6	Soil	Transect A-1 ft	Composite of 3 Grabs	1 ft	A + OVA screen
7A-7	Soil	Transect A-2 ft	Composite of 3 Grabs	2 ft	A + OVA screen
7A-8	Soil	Transect A-3 ft	Composite of 3 Grabs	3 ft	A + OVA screen
7A-9	Soil	Transect B-surf	Composite of 3 Grabs	Surf	A + OVA screen
7A-10	Soil	Transect B-1 ft	Composite of 3 Grabs	1 ft	A + OVA screen
7A-11	Soil	Transect B-2 Ft	Composite of 3 Grabs	2 ft	A + OVA screen
7A-12	Soil	Transect B-3 ft	Composite of 3 Grabs	3 ft	A + OVA screen
7A-13	Soil	Transect C-surf	Composite of 3 Grabs	Surf	A + OVA screen
7A-14	Soil	Transect C-1 ft	Composite of 3 Grabs	1 ft	A + OVA screen
7A-15	Soil	Transect C-2 ft	Composite of 3 Grabs	2 ft	A + OVA screen
7A-16	Soil	Transect C-3 ft	Composite of 3 Grabs	3 ft	A + OVA screen



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SITE 11A

P AREA NORTH

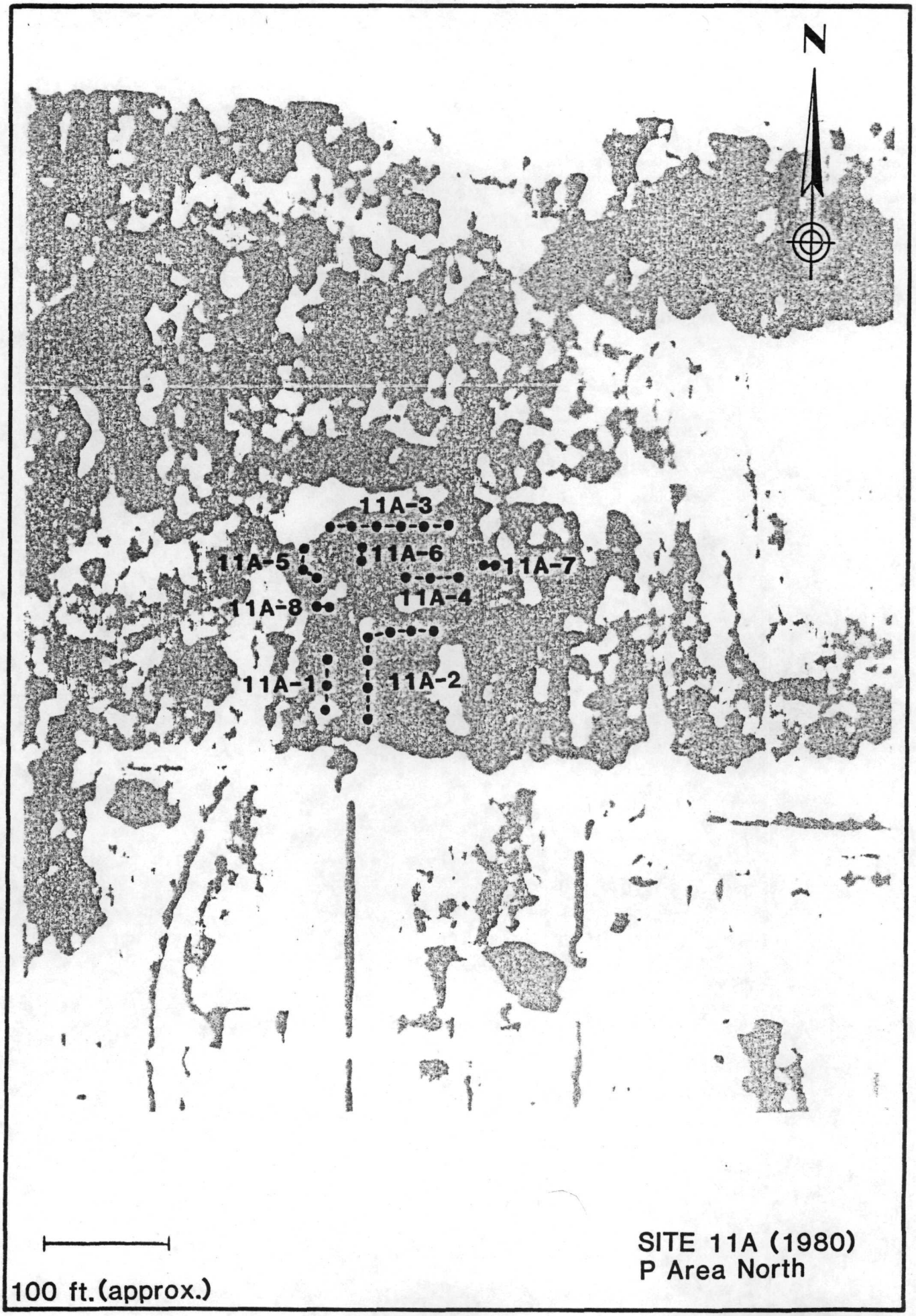
Background

Located outside of the fence north of the Olin P Area is an abandoned L-shaped loading area with connecting covered walkways approximately 100 ft and 85 ft. The central structure contains a loading dock and a steamhouse containing a concrete pit with about 5 ft of clear standing water. An old roadbed runs west and north of the structure and draining swales surround all of the buildings. An abandoned (?) sewer line also runs across the north edge of the site. It has been reported that contaminants were dumped on the ground outside of the building.

Sampling and Analysis Schedule

The proposed surface sampling locations focus on low-lying areas which may have accumulated residues. In addition, four soil areas just outside of doorways are proposed for sampling as indicated below.

I.D.	Matrix	Name	Type	Depth	Analysis Set
11A-1	Sed.	West Swale	Composite of 3 Grabs	0 to 1 ft	A
11A-2	Sed.	East Swale	Composite of 7 Grabs	0 to 1 ft	A
11A-3	Sed.	North Swale 1	Composite of 6 Grabs	0 to 1 ft	A
11A-4	Sed.	North Swale 2	Composite of 3 Grabs	0 to 1 ft	A
11A-5	Soil	Loading Dock	Composite of 3 Grabs	0 to 1 ft	A
11A-6	Soil	North Door	Composite of 2 Grabs	0 to 1 ft	A
11A-7	Soil	East Load Area	Composite of 3 Grabs	0 to 1 ft	A
11A-8	Soil	Steamhouse Door	Composite of 2 Grabs	0 to 1 ft	A



O'BRIEN & GERE ENGINEERS, INC.

SITE 12

AREA 14 LANDFILL

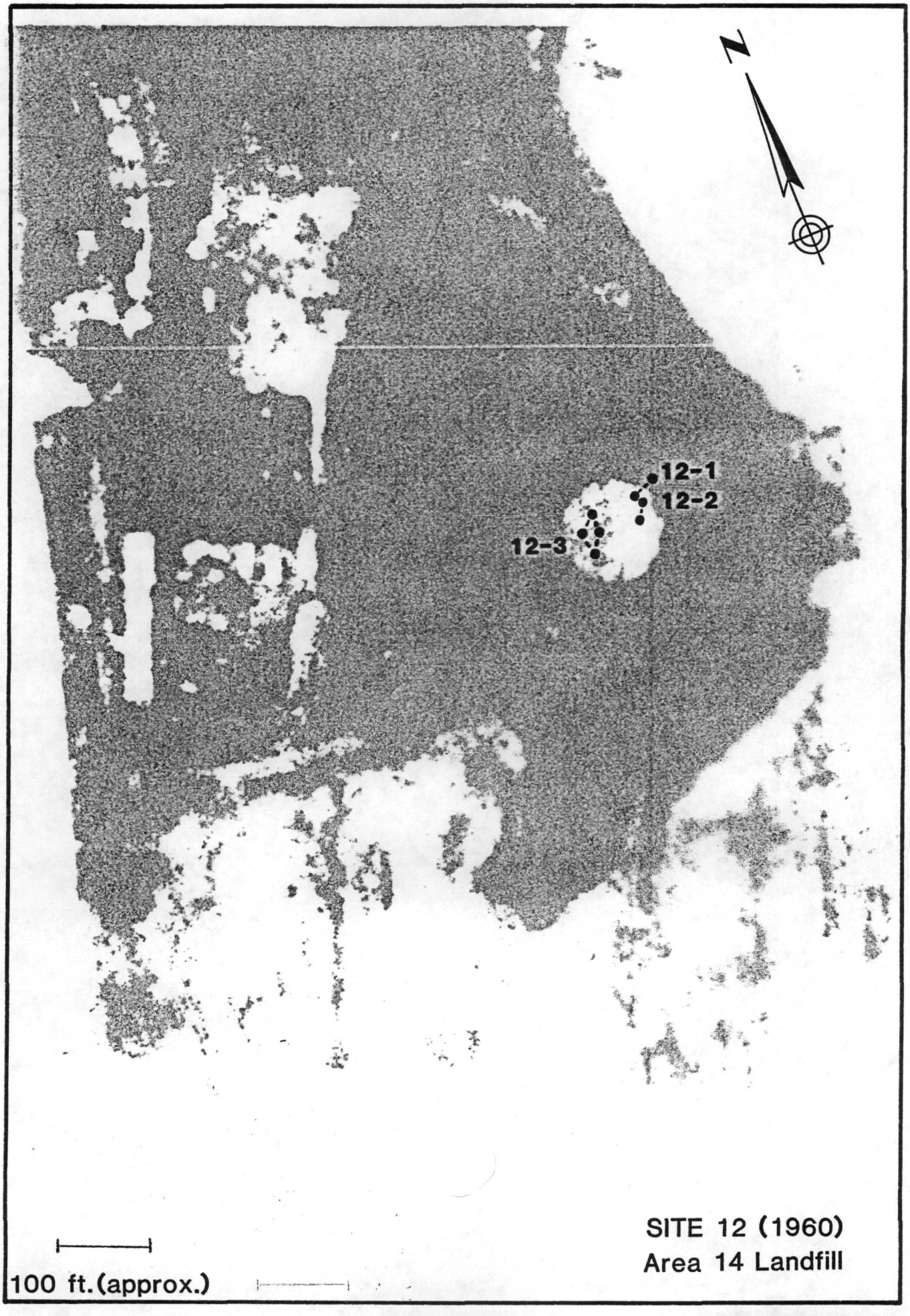
Background

Area 14 was a site of munitions loading activity. Many of the buildings have been abandoned or demolished, but a few industries presently occupy some of the buildings. Historic aerial photos indicated what appeared to be landfill activity in the field east of the presently-occupied buildings. During the site visit the remains of a 100-ft dia circular impoundment were found at this site. The interior of the impoundment is presently overgrown with trees with trunk diameters of 8 to 10 in, indicating the date of the impoundment closure at about 1955 to 1965. The impoundment walls are about 6 ft high and the north wall has been breached to allow drainage to flow from the impoundment to an adjoining field. Several black oily pools are evident within and outside the basin. Other bare patches of black sediment and tars are located around the basin floor.

Sampling and Analysis Schedule

The following Phase I samples are proposed:

I.D.	Matrix	Name	Type	Depth	Analysis Set
12-1	Water	Drainage Channel	Composite of 4 Grabs	Surface	A
12-2	Sed.	Drainage Channel	Composite of 4 Grabs	0 to 1 ft	A
12-3	Soil	Black Residue	Composite of 4 Grabs	0 to 1 ft	D



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SITE 13

AREA 14 CHANGE HOUSE SITE

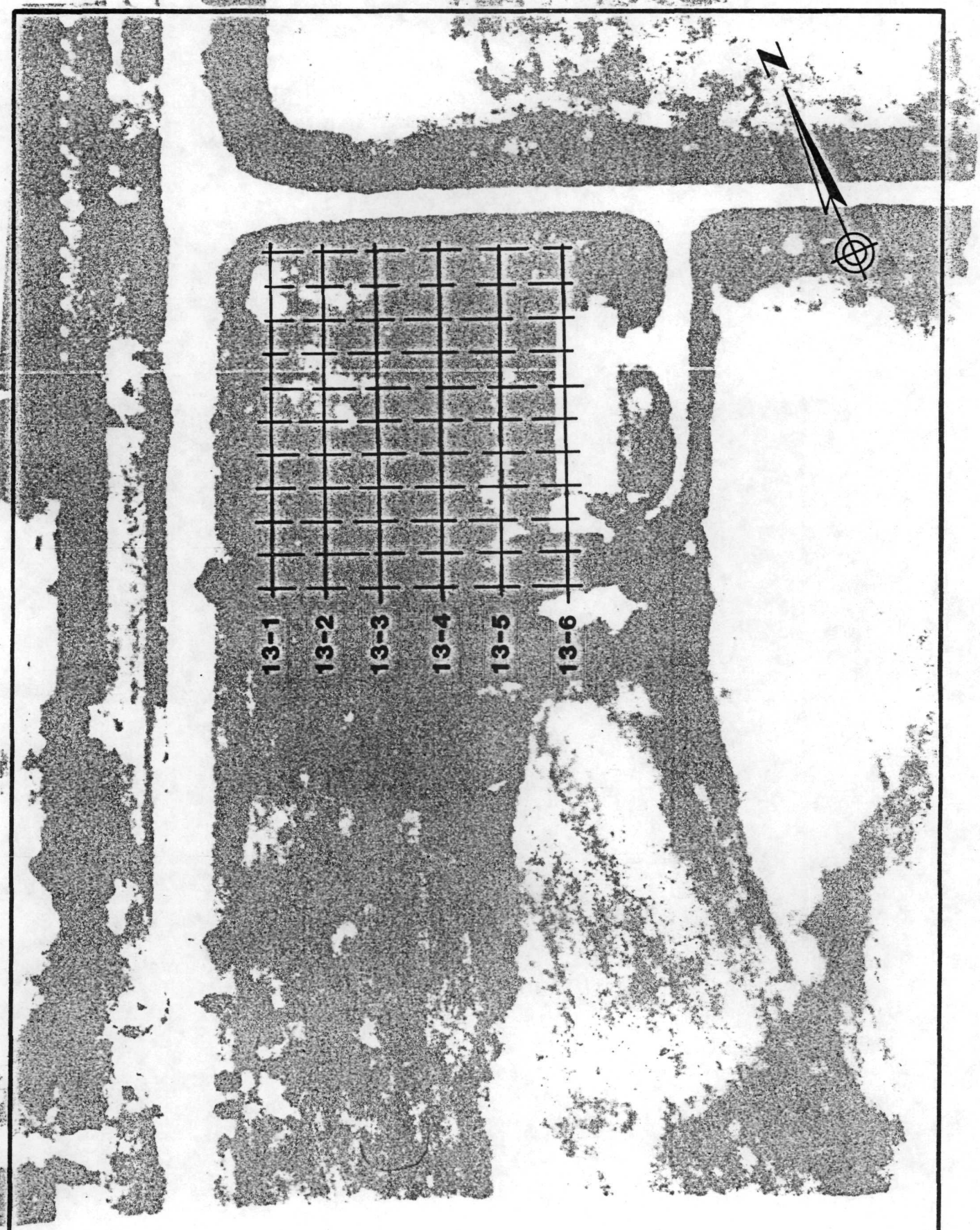
Background

Southeast of the active Diagraph-Bradley buildings on Area 14 was an old building which was recently demolished. Formerly, it was the site of a "Change House" where workers changed their clothing after working in the adjacent bomb-loading buildings. At one time a company named CTI (Chemicals and Technology, Inc.??) manufactured explosives and other chemicals in this building. Other industries may also have occupied this building. The change building was supposedly located across from the bomb-loading building on a plot of land just southeast of the intersection of two roads on the north edge of a big dirt mound. The concrete floor of the change house is under this mound. Aerial photos show another building (no longer present) further east of the corner; field inspection revealed several 1/2-in reinforcing rods imbedded in concrete near the corners of this building.

Sampling and Analysis Schedule

A magnetometer and electromagnetic (EM) survey will be conducted at this site over a 250-ft x 200-ft area. Grid spacing will be on 25-ft centers. Six north-south transect lines will also be established within this area. Ten grab samples of soils will be obtained along each transect. The following Phase I samples are proposed:

I.D.	Matrix	Name	Type	Depth	Analysis Set
13-1	Soil	Transect 1	Composite of 10 Grabs	0 to 1 ft	A
13-2	Soil	Transect 2	Composite of 10 Grabs	0 to 1 ft	A
13-3	Soil	Transect 3	Composite of 10 Grabs	0 to 1 ft	A
13-4	Soil	Transect 4	Composite of 10 Grabs	0 to 1 ft	A
13-5	Soil	Transect 5	Composite of 10 Grabs	0 to 1 ft	A
13-6	Soil	Transect 6	Composite of 10 Grabs	0 to 1 ft	A



100 ft. (approx.)

SITE 13 (1960)
Area 14 Change House Site

O'BRIEN & GERE
ENGINEERS, INC.

SITE 14

AREA 14 SOLVENT STORAGE

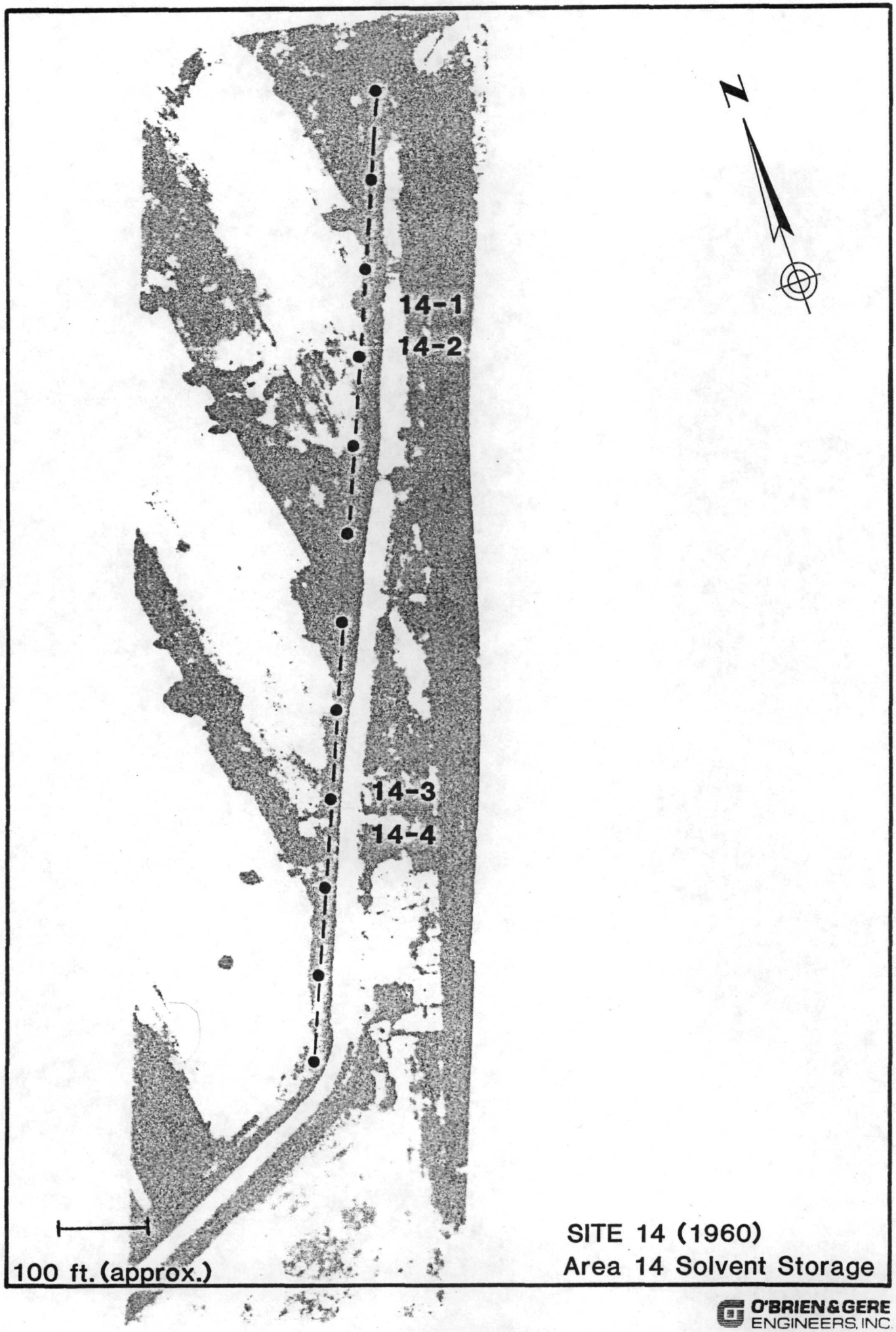
Background

Diagraph-Bradley or Diagraph Marking Systems currently operates within a complex of buildings in Area 14. They produce inks, stencils, stencilboards and marking pens. Linseed oil and various solvents are handled in bulk and in drums here. Some of the bulk solvents noted were: T25 Xylene, T8 Diacetone Alcohol, T9 Diethylene Glycol, and T18 Methyl Cellosolve. Several compressed gas cylinders are also present. At least two drum storage areas containing 50 to 200 drums were also noted. Spill containment facilities are minimal. A drainage ditch runs north parallel to the road west of the buildings. Process water from the Diagraph-Bradley buildings enters this ditch from a standpipe.

Sampling and Analysis Schedule

Traces of solvent spillage will be evaluated by sampling waters and sediments within the ditch west of the Diagraph-Bradley buildings. The following Phase I samples are proposed:

I.D.	Matrix	Name	Type	Depth	Analysis Set
14-1	Water	Ditch North	Composite of 6 Grabs	Surface	A
14-2	Sed.	Ditch North	Composite of 6 Grabs	0 to 1 ft	A
14-3	Water	Ditch South	Composite of 6 Grabs	Surface	A
14-4	Sed.	Ditch South	Composite of 6 Grabs	0 to 1 ft	A



SITE 14 (1960)
Area 14 Solvent Storage

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SITES 15 AND 16
AREA 7 PLATING POND
AREA 7 INDUSTRIAL SITE

Background

Area 7 contains a complex of 33 identical buildings which have been used for a variety of industrial purposes during the past 40 years. Each of the six rows of buildings was previously served by a railroad siding.

Within a wooded rise to the south is located a small pond (approximately 50 ft x 30 ft) which is bermed about five ft above the current water level. The current water depth is estimated to be about four ft. It is claimed that this pond was used to receive plating wastewaters from Olin operations which were located in this area at one time. PCBs, lead and other heavy metals may be of concern here.

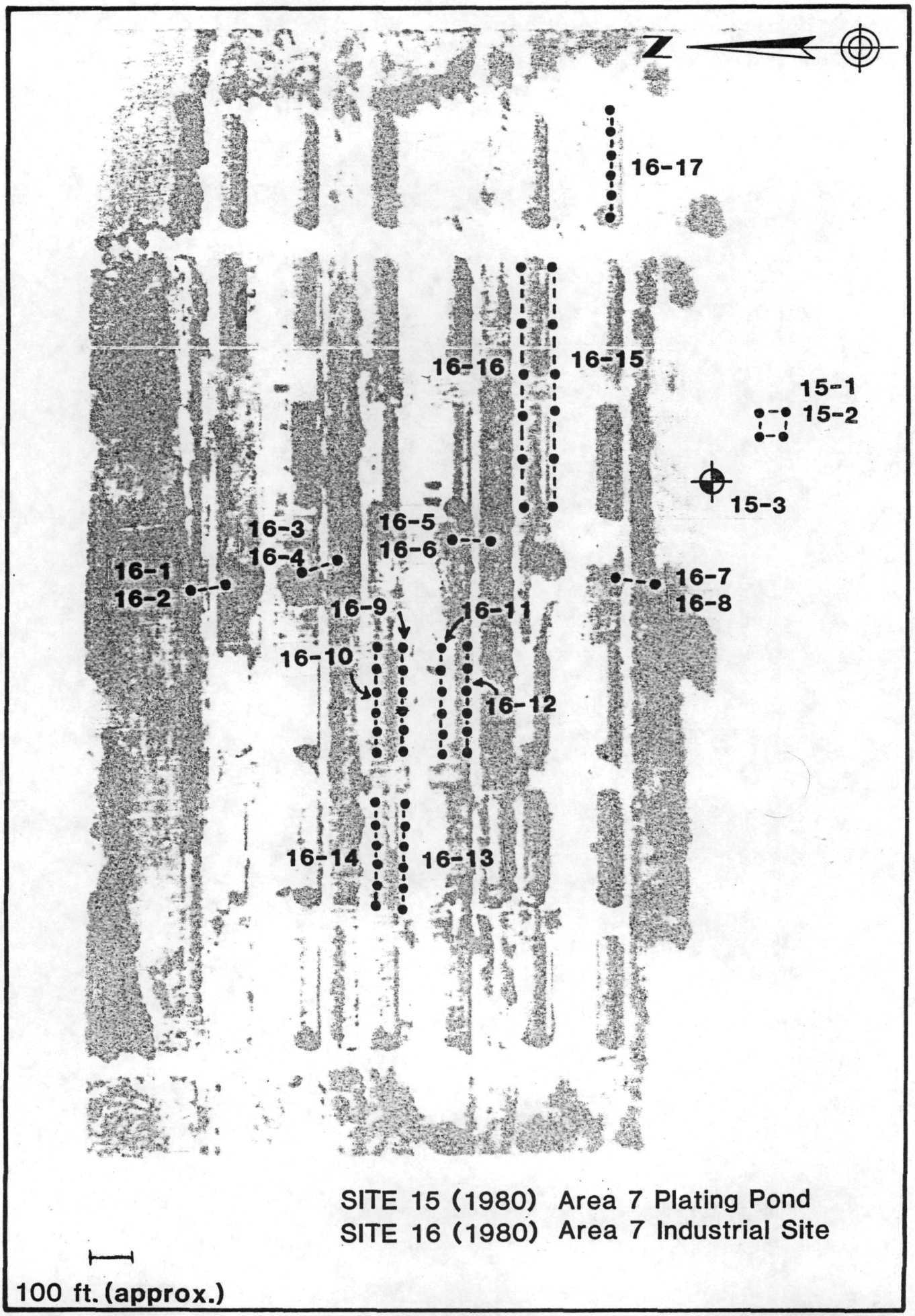
Many of the buildings on the Area 7 site are used for dry warehousing purposes. However, two specific locations have been specified for sampling. Buildings 3-4, 3-5, and 4-4 are used by Pennzoil for waste oil recovery and recycling operations. Black residues are noticeable around some of these buildings. Buildings 5-2 and 5-3 are used by a refurbisher of mining machinery. Black residues are also evident around these buildings. A drainage channel runs from south to north through the center of the site.

Sampling and Analysis Schedule

Waters and sediments will be sampled within the Area 7 Plating Pond. In addition, a single groundwater monitor well will be installed downgradient of the Plating Pond and sampled for any evidence of groundwater contamination.

Composite soil samples will be obtained from around five of the building perimeters within Area 7. In addition, waters and sediments will be sampled from the drainage channel which bisects the buildings. The following Phase I samples are proposed:

I.D.	Matrix	Name	Type	Depth	Analysis Set
15-1	Water	Plating Pond	Composite of 4 Grabs	Surface	A
15-2	Sed.	Plating Pond	Composite of 4 Grabs	0 to 1 ft	A
15-3	Water	Monitor Well	Single Sampling	Bailer	A
16-1	Water	Ditch No. 1	Composite of 2 Grabs	Surface	A
16-2	Sed.	Ditch No. 1	Composite of 2 Grabs	0 to 1 ft	A
16-3	Water	Ditch No. 2	Composite of 2 Grabs	Surface	A
16-4	Sed.	Ditch No. 2	Composite of 2 Grabs	0 to 1 ft	A
16-5	Water	Ditch No. 3	Composite of 2 Grabs	Surface	A
16-6	Sed.	Ditch No. 3	Composite of 2 Grabs	0 to 1 ft	A
16-7	Water	Ditch No. 4	Composite of 2 Grabs	Surface	A
16-8	Sed.	Ditch No. 4	Composite of 2 Grabs	0 to 1 ft	A
16-9	Soil	Bldg 3-4 Front	Composite of 6 Grabs	0 to 1 ft	D
16-10	Soil	Bldg 3-4 Back	Composite of 6 Grabs	0 to 1 ft	A
16-11	Soil	Bldg 3-5 Front	Composite of 6 Grabs	0 to 1 ft	A
16-12	Soil	Bldg 3-5 Back	Composite of 6 Grabs	0 to 1 ft	A
16-13	Soil	Bldg 4-4 Front	Composite of 6 Grabs	0 to 1 ft	A
16-14	Soil	Bldg 4-4 Back	Composite of 6 Grabs	0 to 1 ft	A
16-15	Soil	Bldg 5-2&3 Front	Composite of 6 Grabs	0 to 1 ft	D
16-16	Soil	Bldg 5-2&3 Back	Composite of 6 Grabs	0 to 1 ft	A
16-17	Soil	Bldg 6-1 Control	Composite of 6 Grabs	0 to 1 ft	A



SITE 15 (1980) Area 7 Plating Pond
SITE 16 (1980) Area 7 Industrial Site

O'BRIEN & GERE ENGINEERS, INC.

SITE 17

JOB CORPS LANDFILL

Background

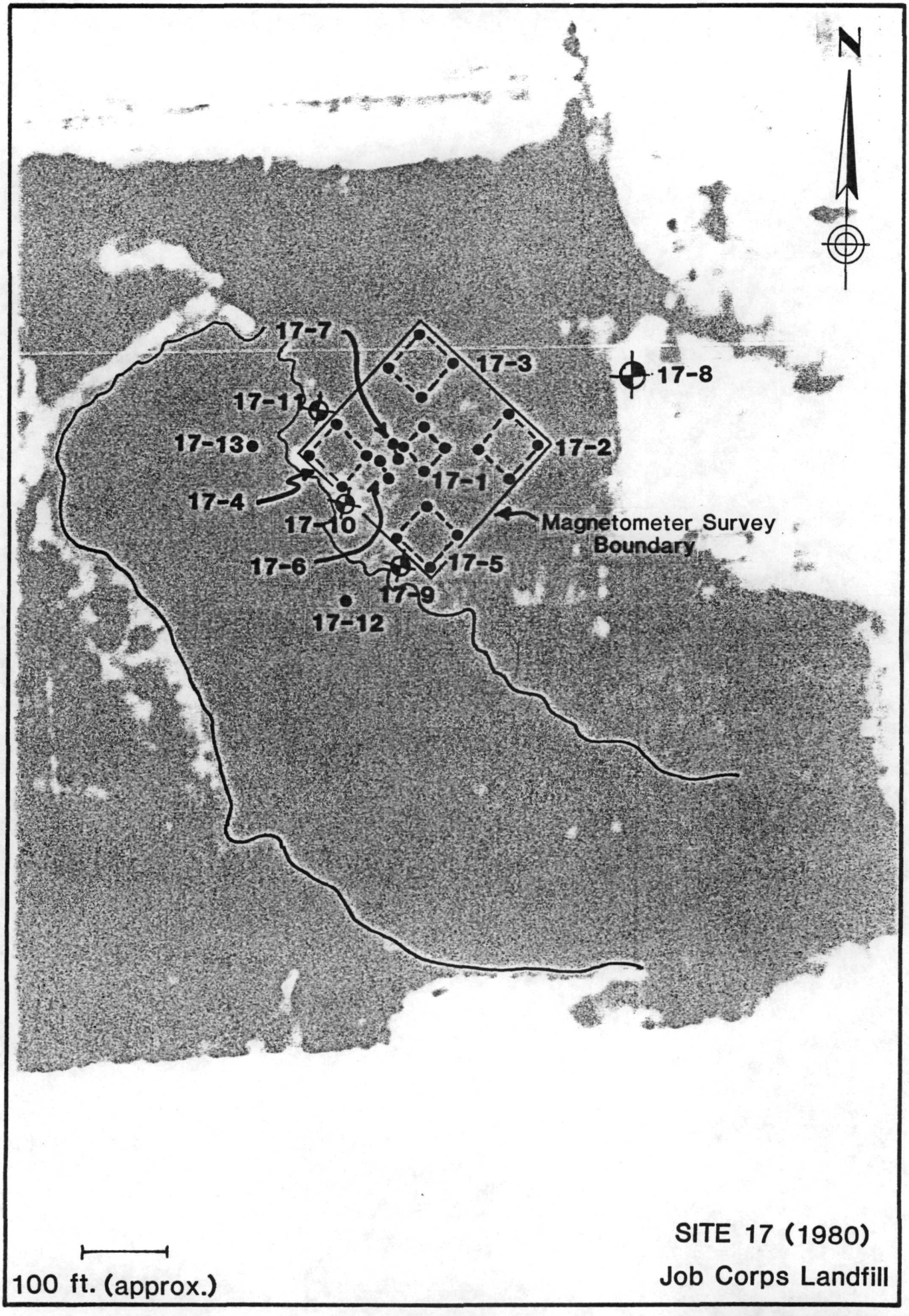
Northeast of the Refuge Waterworks is a small (approximately 10 acre) pond created by Job Corps workers in the mid-1960's. Attention has recently been brought to this pond because as many as thirty or more geese carcasses have been found floating on the water or littering the shores. Some of these carcasses have been relatively fresh while others were in various state of decay. The Fish and Wildlife Service has completed extensive analyses of these carcasses and has ruled out a variety of potential chemical causes. A definite conclusion has not yet been reached.

The "Job Corps" landfill was discovered while investigating the geese kills. It is located within a wooded area to the north and adjoining the pond and covers an area of perhaps an acre of more. It appears to be mainly surface litter dumped in spots and perhaps spread around, although deeper spots cannot be ruled out. Many of the surface articles appear to be connected with food preparation, e.g. instutional-size food cans, and a variety of bottles. The bottle styles and labels suggest a date of the mid-1950's, which was consistent with a 1956 Illinois automobile license plate also found. Many of the debris piles are overgrown by thick brush. Two bare patches (less than 6-ft diameter each) were located among the debris. Mica flakes and small electrical contacts were found in one of these. It is claimed that small electrical capacitors were also found here, but none were noted during this site visit. Probing with a trowel revealed no further debris beneath the top inch of soil.

Sampling and Analysis Schedule

A magnetometer and electromagnetic (EM) survey will be conducted over a 200 ft x 200 ft area on 10 ft x 10 ft grid spacings. Soil samples will be composited within five 50-ft sided square grids within the landfill area. In addition, soil samples will be obtained from each of the two bare patches. Depth of soil samples is planned to 1 ft, but contingent upon the results of the geophysical surveys. Four shallow wells may be placed (3 downgradient and 1 upgradient) and sampled. These wells may be installed and sampled during Phase II after the depth of fill activity has been determined. Two surface water samples from the pond also will be analyzed. The following Phase I samples are proposed:

I.D.	Matrix	Name	Type	Depth	Analysis Set
17-1	Soil	Soil Grid 1	Composite of 5 Grabs	0 to 1 ft	A
17-2	Soil	Soil Grid 2	Composite of 5 Grabs	0 to 1 ft	A
17-3	Soil	Soil Grid 3	Composite of 5 Grabs	0 to 1 ft	D
17-4	Soil	Soil Grid 4	Composite of 5 Grabs	0 to 1 ft	A
17-5	Soil	Soil Grid 5	Composite of 5 Grabs	0 to 1 ft	A
17-6	Soil	Bare Patch 1	Composite of 2 Grabs	0 to 1 ft	D
17-7	Soil	Bare Patch 2	Composite of 2 Grabs	Surface	A
17-8	Water	Well 17-1	Single Sampling	Bailer	A
17-9	Water	Well 17-2	Single Sampling	Bailer	A
17-10	Water	Well 17-3	Single Sampling	Bailer	A
17-11	Water	Well 17-4	Single Sampling	Bailer	A
17-12	Water	Pond No. 1	Single Sampling	Surface	A
17-13	Water	Pond No. 2	Single Sampling	Surface	A



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SITE 18

AREA 13 LOADING PLATFORM

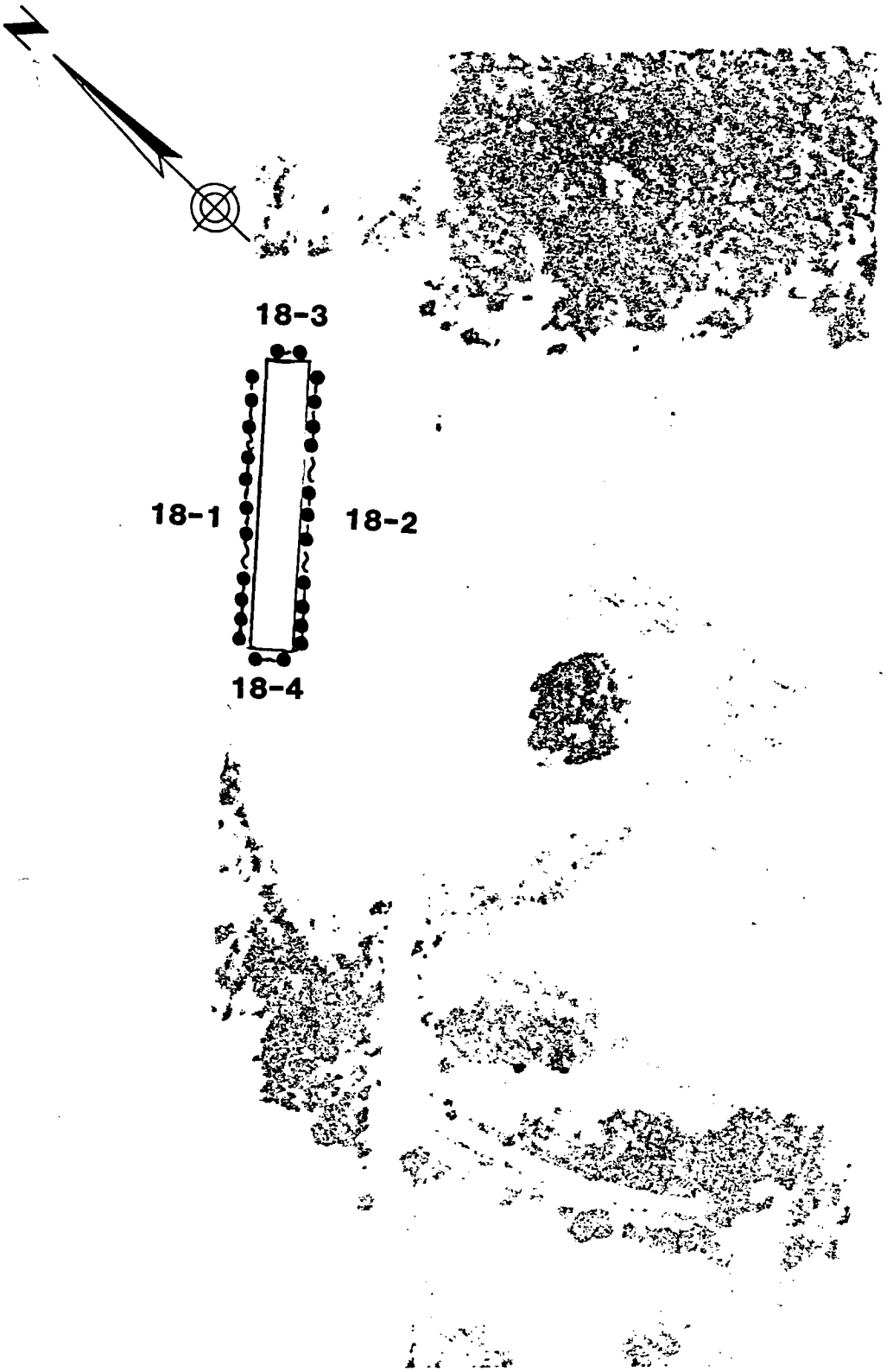
Background

On the northwest end of the Area 13 munitions storage bunkers is a concrete loading platform adjacent to the abandoned and dismantled rail line. It is reported that munitions-type chemicals were dumped off the platform. The site inspection indicated that the elevated concrete loading dock is about 235 ft long by 10 ft wide and about 5 ft high. The dock is supported on concrete posts spaced 9 ft apart. The northwest side contains stone bedding (probably from the oil railroad bed) with a number of small areas of ponded water. No unusual vegetation changes were detected. The only unusual item was a pile of dirt and stone rubble off the west end of the dock with a rusted drum shell nearby.

Sampling and Analysis Schedule

Samples of soil will be obtained from each end and along both sides of the loading dock according to the following schedule:

I.D.	Matrix	Name	Type	Depth	Analysis Set
18-1	Soil	Loading Dock N	Composite of 20 Grabs	0 to 1 ft	A
18-2	Soil	Loading Dock S	Composite of 20 Grabs	0 to 1 ft	A
18-3	Soil	Loading Dock E	Composite of 2 Grabs	0 to 1 ft	A
18-4	Soil	Loading Dock W	Composite of 2 Grabs	0 to 1 ft	A



SITE 18 (1980) Area 13 Loading Platform

100 ft. (approx.)

O'BRIEN & GERE ENGINEERS, INC.

SITE 19

AREA 13 BUNKER 1-3

Background

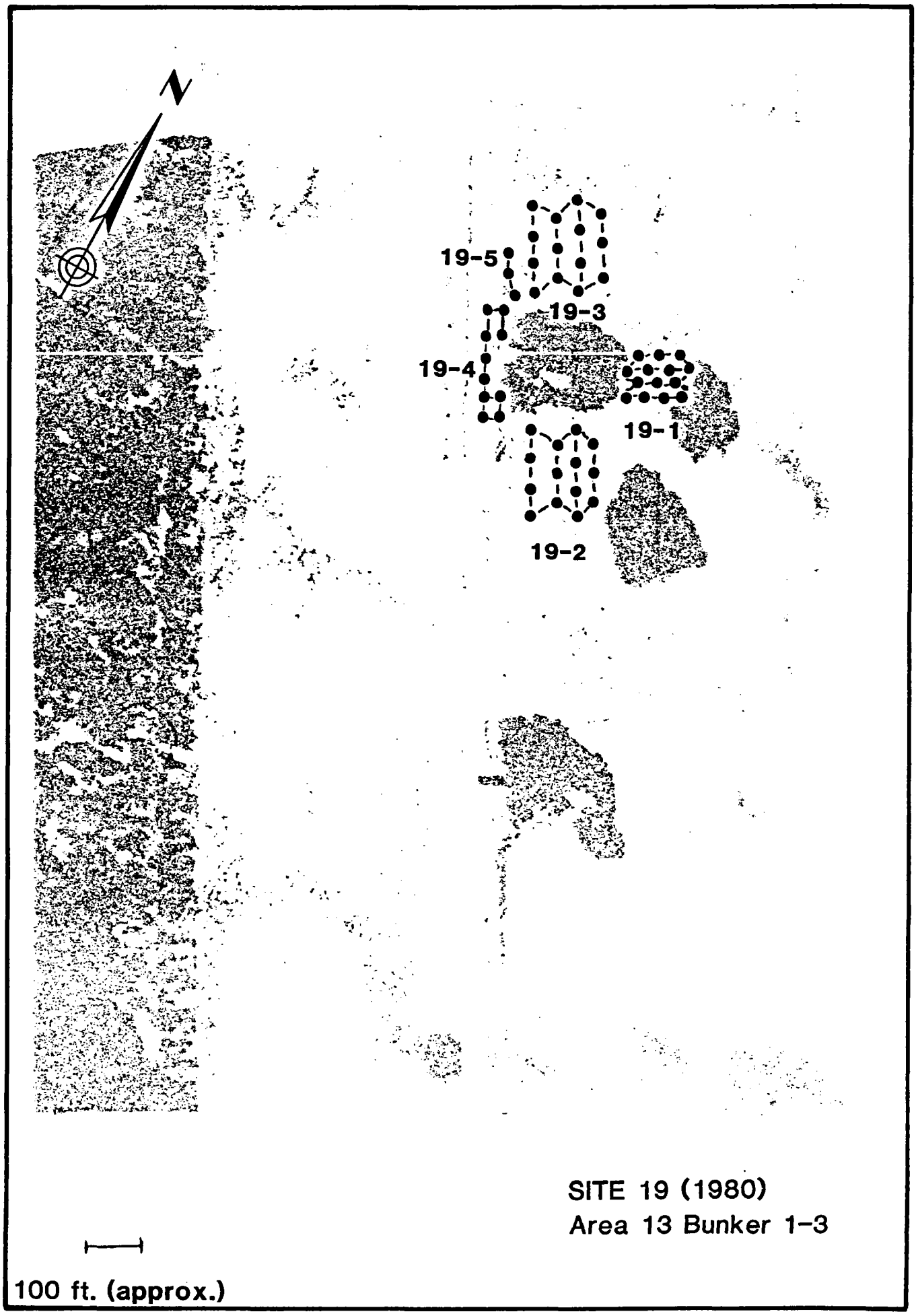
Area 13 contains approximately 85 bunkers which were originally built for storage of 500-lb bombs. Most of them still contain explosives, leased mainly to Olin and U.S. Powder. Agricultural fields are cultivated between the bunkers. Formerly, they were fruit orchards.

It has been reported that chemicals were poured out near Bunker 1-3, probably in the field next to it. A site inspection did not reveal any significant signs of impact. Evidence of fill activity (scattered red bricks) is widespread. An L-shaped area of brown vegetation difference was noted to the west side of the bunker.

Sampling and Analysis Schedule

Soil samples will be composited within three 50-ft sided square grids adjacent to Bunker 1-3. In addition, ten soil samples will be composited from the front side of the bunker and a composite will be obtained from the brown patch to the northwest. The following Phase I samples are proposed:

I.D.	Matrix	Name	Type	Depth	Analysis Set
19-1	Soil	Soil Grid NE	Composite of 14 Grabs	0 to 1 ft	A
19-2	Soil	Soil Grid SE	Composite of 14 Grabs	0 to 1 ft	A
19-3	Soil	Soil Grid NW	Composite of 14 Grabs	0 to 1 ft	A
19-4	Soil	Soil Grid Front	Composite of 10 Grabs	0 to 1 ft	A
19-5	Soil	Br. Patch Transct	Composite of 3 Grabs	0 to 1 ft	A



SITE 19 (1980)
Area 13 Bunker 1-3

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SITE 20

D AREA SOUTH

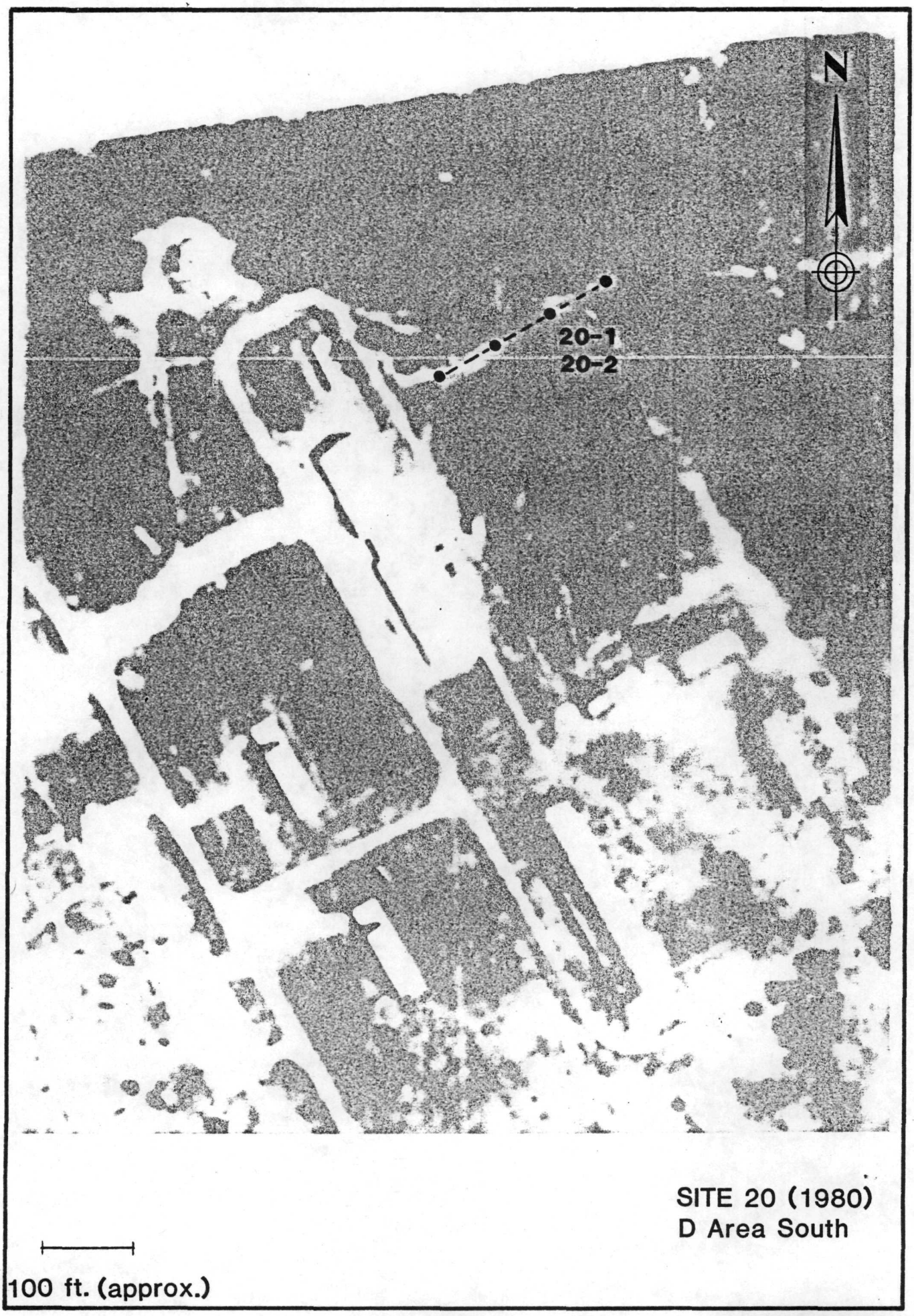
Background

An abandoned building is located within the fenced southeastern end of the Olin D Complex. It was reported that chemicals were dumped here. A drainage swale originating at the building runs east outside of the fence. A four-in pipe (dripping) extends from the Olin Area under the fence and discharges to this ditch. A slight sheen was noticeable on the surface water in pooled areas of the ditch.

Sampling and Analysis Schedule

Waters and sediments will be composited from the ditch in accordance with the following Phase I schedule:

I.D.	Matrix	Name	Type	Depth	Analysis Set
20-1	Water	D South	Composite of 4 Grabs	Surface	A
20-2	Sed.	D South	Composite of 4 Grabs	0 to 1 ft	A



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SITE 21

SOUTHEAST CORNER FIELD

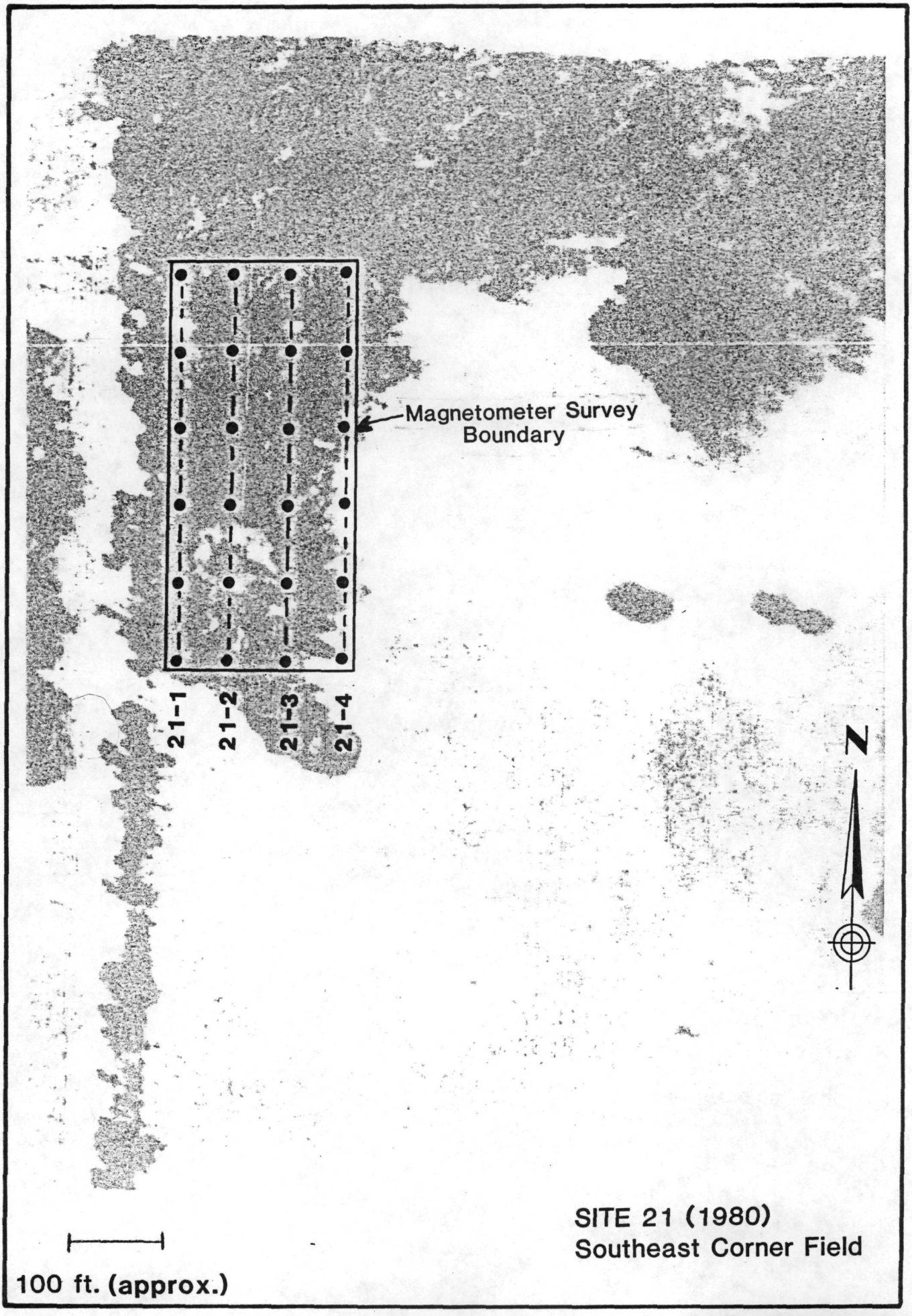
Background

At the southeast corner of the refuge is a field which is thought to be the site of a very old landfill. A pile of concrete pieces, possibly from an old bridge, is located immediately inside the fence. The topography gradually slopes to the south and east with a swampy drainage ditch at the bottom of the slope. No other evidence of debris could be found. Trees as large as 24-in in diameter suggest that the area has not seen any soil-disturbing activity within the past 60 to 70 years.

Sampling and Analysis Schedule

A magnetometer and electromagnetic (EM) survey will be conducted within the 200-ft x 125-ft area along four north-south transects. Soil composites will also be taken along these same transects as follows:

I.D.	Matrix	Name	Type	Depth	Analysis Set
21-1	Soil	Transect 1	Composite of 6 Grabs	0 to 1 ft	A
21-2	Soil	Transect 2	Composite of 6 Grabs	0 to 1 ft	A
21-3	Soil	Transect 3	Composite of 6 Grabs	0 to 1 ft	A
21-4	Soil	Transect 4	Composite of 6 Grabs	0 to 1 ft	A



SITE 21 (1980)
Southeast Corner Field

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SITE 22

OLD REFUGE SHOP

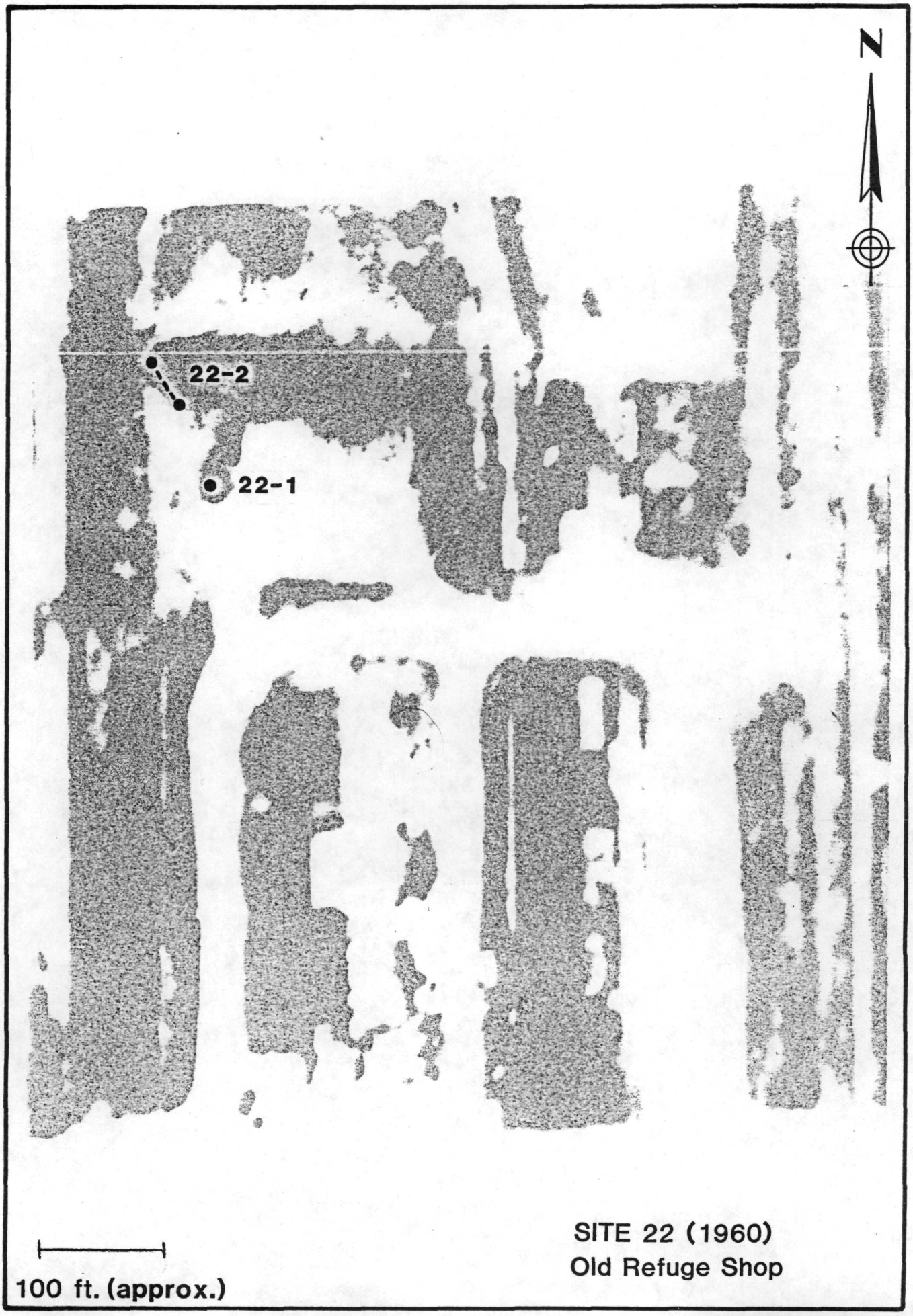
Background

North of the refuge along Wolf Creek Road is the old refuge headquarters, now leased by Diagraph Bradley. Behind this building is located the old shop area of the refuge. Pine poles were treated here with pentachlorphenol and shipped to various spots around the country. Outside the fence to the north is a small pool which receives drainage from the old shop area. The pool contains a green-yellow scum and drains through the woods to the northwest.

Sampling and Analysis Schedule

The following samples will be obtained as part of Phase I:

I.D.	Matrix	Name	Type	Depth	Analysis Set
22-1	Water	Pool Water	Single Grab	Surface	A
22-2	Sed.	Stream Sed.	Composite of 2 Grabs	0 to 1 ft	A



SITE 22 (1960)
Old Refuge Shop

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SITE 24

PEPSI-WEST

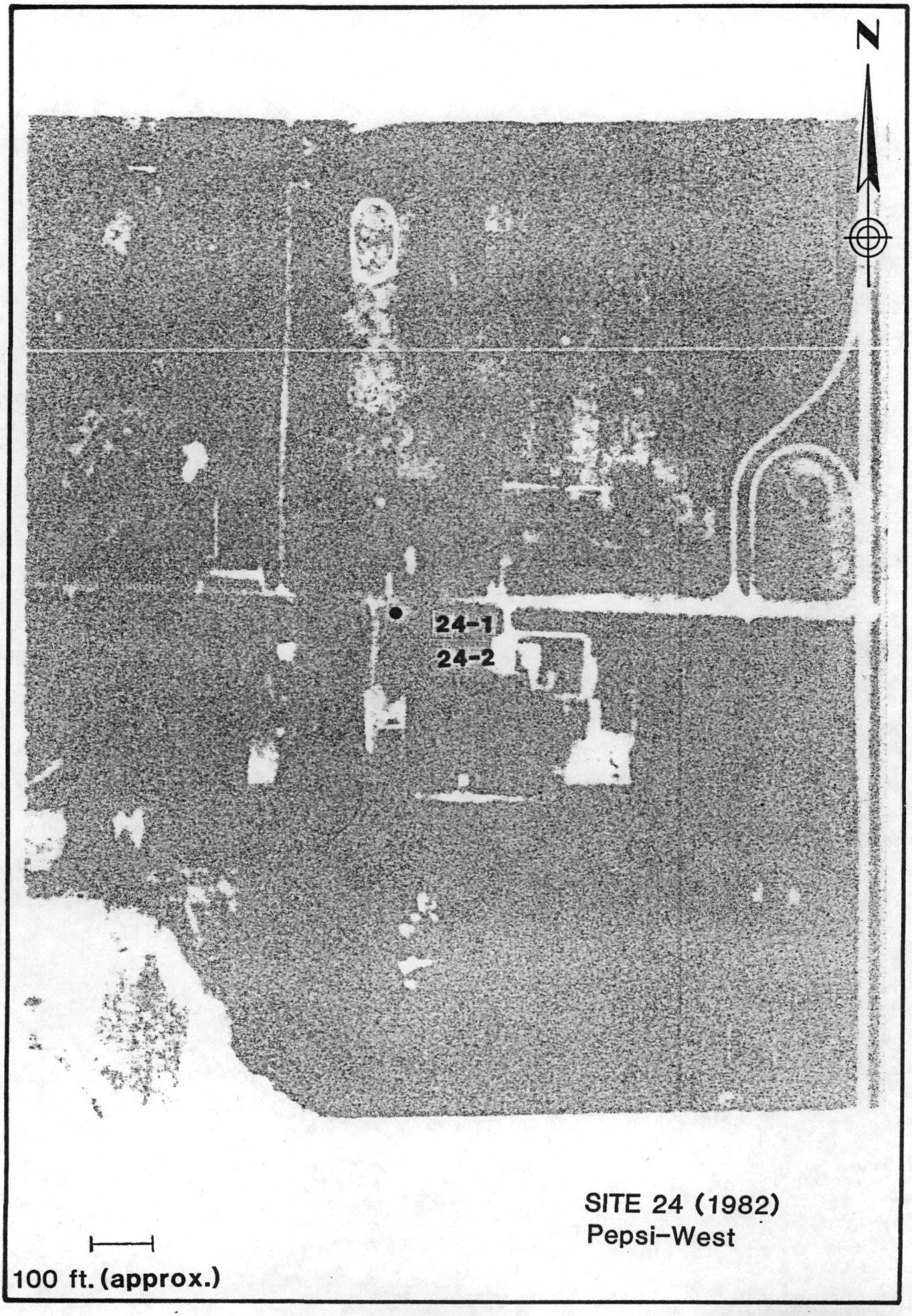
Background

The Pepsi Cola Bottling Company in Marion could potentially discharge to Crab Orchard Creek. It is not known whether the City or State monitor environmental activities here. A site inspection indicated that it was unlikely that discharges issued directly south to the Creek, since the entire south end of the property rises 4 to 8 ft in elevation above the parking lot. Drainage ditches, however, were located to the north adjacent to the street. These probably receive surface runoff only.

Sampling and Analysis Schedule

A single grab sample will be obtained from the north ditch during Phase I.

I.D.	Matrix	Name	Type	Depth	Analysis Set
24-1	Water	Pepsi-West	Single Grab	Surface	A
24-1	Sed.	Pepsi-West	Single Grab	0 to 1 ft	A



SITE 24 (1982)
Pepsi-West

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SITE 25

CRAB ORCHARD CREEK AT MARION LANDFILL

Background

The old Marion landfill is off Old Creal Springs Road and directly abuts Crab Orchard Creek. It has apparently been inactive for a number of years. A visible face of trash can be seen by travelling upstream several hundred yards from the road. Near to this is a small pond (approximately 3/4 acre).

Sampling and Analysis Schedule

The following samples are proposed for Phase I:

I.D.	Matrix	Name	Type	Depth	Analysis Set
25-1	Water	COC Downstream	Composite of 3 Grabs	Surface	A
25-2	Sed.	COC Downstream	Composite of 3 Grabs	0 to 1 ft	D
25-3	Water	COC Upstream	Composite of 3 Grabs	Surface	A
25-4	Sed.	COC Upstream	Composite of 3 Grabs	0 to 1 ft	A
25-5	Water	LF Pond	Composite of 3 Grabs	Surface	A
25-6	Sed.	LF Pond	Composite of 3 Grabs	0 to 1 ft	A

SITES 26 AND 27

CRAB ORCHARD CREEK BELOW MARION STP

CRAB ORCHARD CREEK BELOW 157 DREDGE AREA

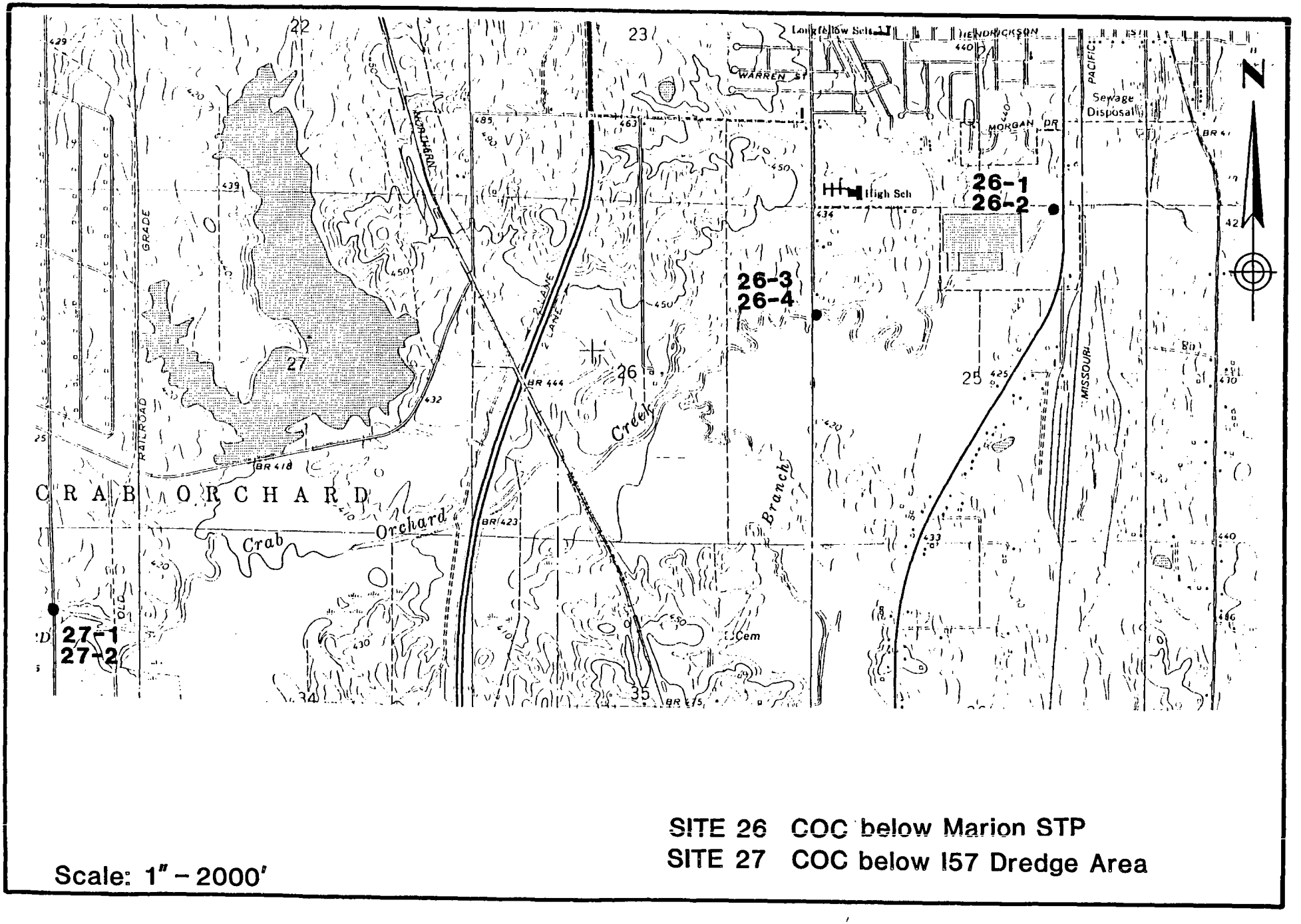
Background

The Marion sewage treatment plant discharges to Crab Orchard Creek somewhere upstream of Court Street. A number of samples downstream from the Marion STP are scheduled to assess the quality of various stretches of Crab Orchard Creek.

Sampling and Analysis Schedule

The following samples are schedule for Phase I:

I.D.	Matrix	Name	Type	Depth	Analysis Set
26-1	Water	COC at Court St.	Composite of 3 Grabs	Surface	A
26-2	Sed.	COC at Court St.	Composite of 3 Grabs	0 to 1 ft	A
26-3	Water	COC at S. Carbon	Composite of 3 Grabs	Surface	A
26-4	Sed.	COC at S. Carbon	Composite of 3 Grabs	0 to 1 ft	A
27-1	Water	COC at Chammness	Composite of 3 Grabs	Surface	A
27-2	Sed.	COC at Chammness	Composite of 3 Grabs	0 to 1 ft	D



SITE 26 COC below Marion STP
SITE 27 COC below I57 Dredge Area

Scale: 1" – 2000'

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SITE 28

WATER TOWER LANDFILL

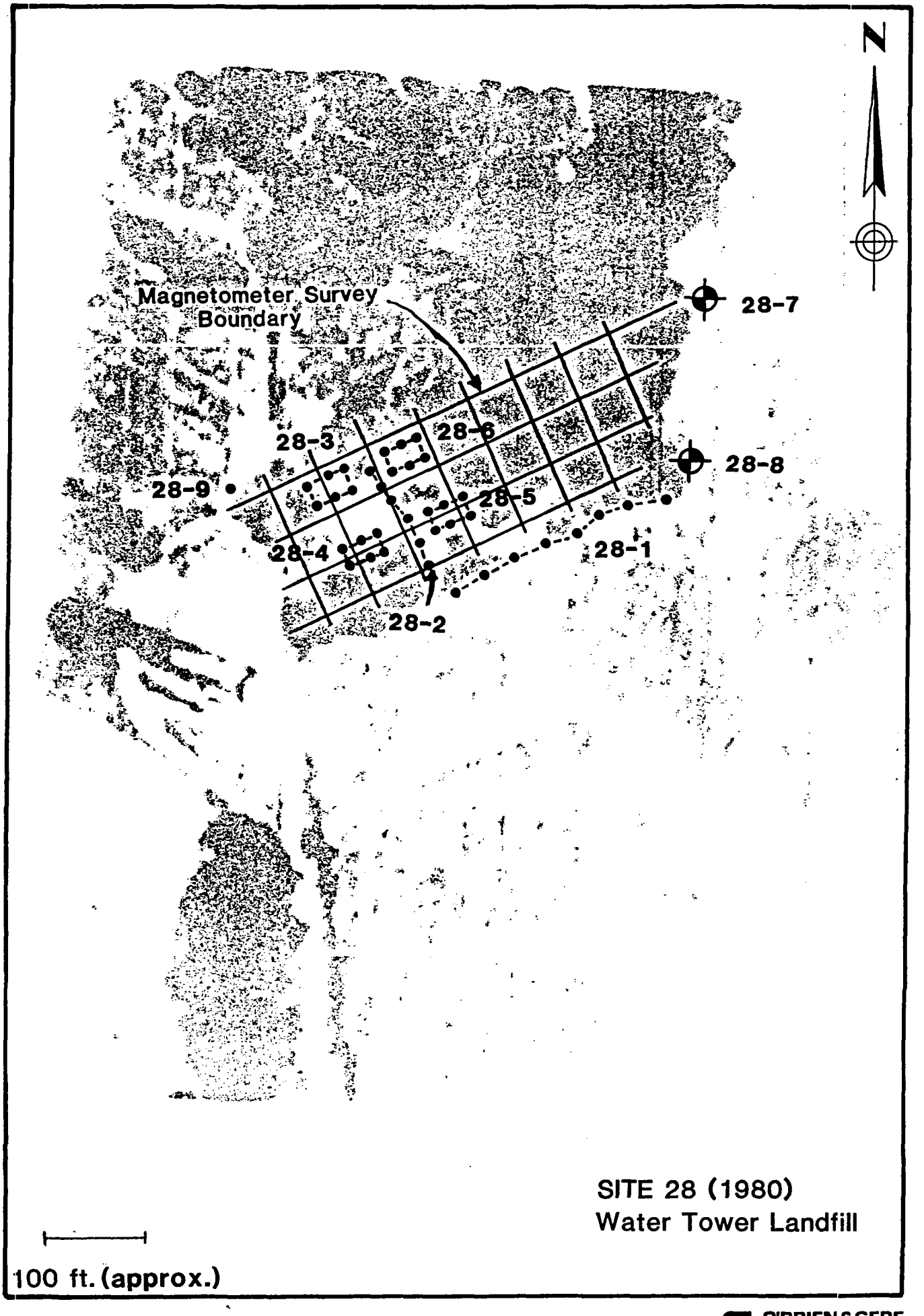
Background

Aerial photos indicate landfilling activities adjacent to the water tower near Areas 7 and 14. These activities are not visually apparent today. The sloping face northeast of the water tower is heavily overgrown with briars and rutted with several major gullies. Only a small amount of refuse is evident on this slope. A previous soil sample taken in this area showed 800 ppm lead concentration. More activity is evident in the woods at the bottom of the slope. A number of rusted drums, metal parts and tar residues can be found here. Standing water in the main drainage gully shows a slight sheen on the surface. Several small mounds are within the woods and a larger mound is located at the top of the hill.

Sampling and Analysis Schedule

Magnetometer and electromagnetic (EM) transect lines will be established along and transverse to the slope to detect locations of subsurface debris. Soil samples will be obtained from the main gully and main transverse gully in addition to four rectangular sampling grids. Six additional grab samples will be obtained at the discretion of the field geologist. Two shallow groundwater monitor wells will be installed at the foot of the hill. The following Phase I sampling program is schedule:

I.D.	Matrix	Name	Type	Depth	Analysis Set
28-1	Soil	Main Gully	Composite of 8 Grabs	0 to 1 ft	D
28-2	Soil	Trans. Gully	Composite of 6 Grabs	0 to 1 ft	A
28-3	Soil	Soil Grid 1	Composite of 6 Grabs	0 to 1 ft	A
28-4	Soil	Soil Grid 2	Composite of 6 Grabs	0 to 1 ft	A
28-5	Soil	Soil Grid 3	Composite of 6 Grabs	0 to 1 ft	A
28-6	Soil	Soil Grid 4	Composite of 6 Grabs	0 to 1 ft	A
28-7	Water	Well 28-1	Single Grab	Bailer	A
28-8	Water	Well 28-2	Single Grab	Bailer	A
28-9	Soil	Xtra 1	Grab	0 to 1 ft	A
28-10	Soil	Xtra 2	Grab	0 to 1 ft	A
28-11	Soil	Xtra 3	Grab	0 to 1 ft	A
28-12	Soil	Xtra 4	Grab	0 to 1 ft	A
28-13	Soil	Xtra 5	Grab	0 to 1 ft	A
28-14	Soil	Xtra 6	Grab	0 to 1 ft	A



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SITE 29

FIRE STATION LANDFILL

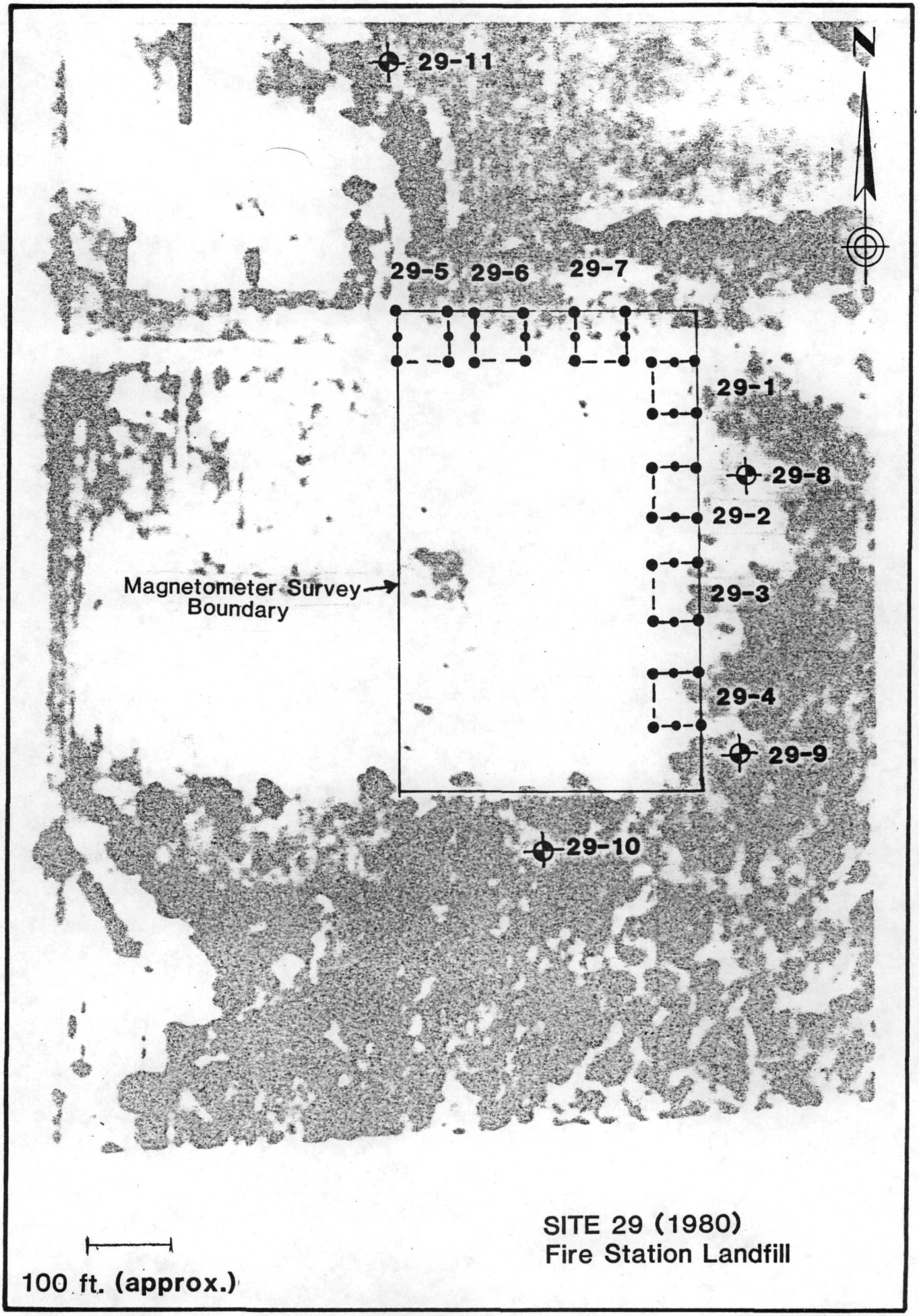
Background

Located southwest of the refuge fire station is a large field which was used for storage of mining machinery until several years ago. The northern and western edges of this field show evidence of a large dump site. Debris is evident on the face which drops 4-5 ft. to a swampy area to the west. Previous sampling near an evergreen tree on the north side showed lead concentrations of 553 ppm. A slight sheen is noted in spots within the swamp. Most of the debris consists of concrete, metal, wire and other machinery-related items. It was reported that Olin dumped heavily here and there once was a very hot fire. Ignitable magnesium is suspected to be in the fill. An empty 30-gal drum labelled "Magnesium Powder" was found along the south portion of the eastern face.

Sampling and Analysis Schedule

A magnetometer and electromagnetic (EM) survey will be conducted over the 350-ft x 300-ft eastern end of the field on grid spacings of 20 ft. Four rectangular soil compositing grids will be established along the eastern face and three similar grids will be established on the northern face. In addition, four groundwater monitor wells will be located (three downgradient and one upgradient). Sampling for Phase I will be as follows:

I.D.	Matrix	Name	Type	Depth	Analysis Set
29-1	Soil	E. Face 1	Composite of 12 Grabs	0 and 1 ft	A
29-2	Soil	E. Face 2	Composite of 12 Grabs	0 and 1 ft	A
29-3	Soil	E. Face 3	Composite of 12 Grabs	0 and 1 ft	D
29-4	Soil	E. Face 4	Composite of 12 Grabs	0 and 1 ft	A
29-5	Soil	N. Face 1	Composite of 12 Grabs	0 and 1 ft	A
29-6	Soil	N. Face 2	Composite of 12 Grabs	0 and 1 ft	D
29-7	Soil	N. Face 3	Composite of 12 Grabs	0 and 1 ft	A
29-8	Water	Well 29-1	Single Grab	Bailer	A
29-9	Water	Well 29-2	Single Grab	Bailer	A
29-10	Water	Well 29-3	Single Grab	Bailer	A
29-11	Water	Well 29-4	Single Grab	Bailer	A



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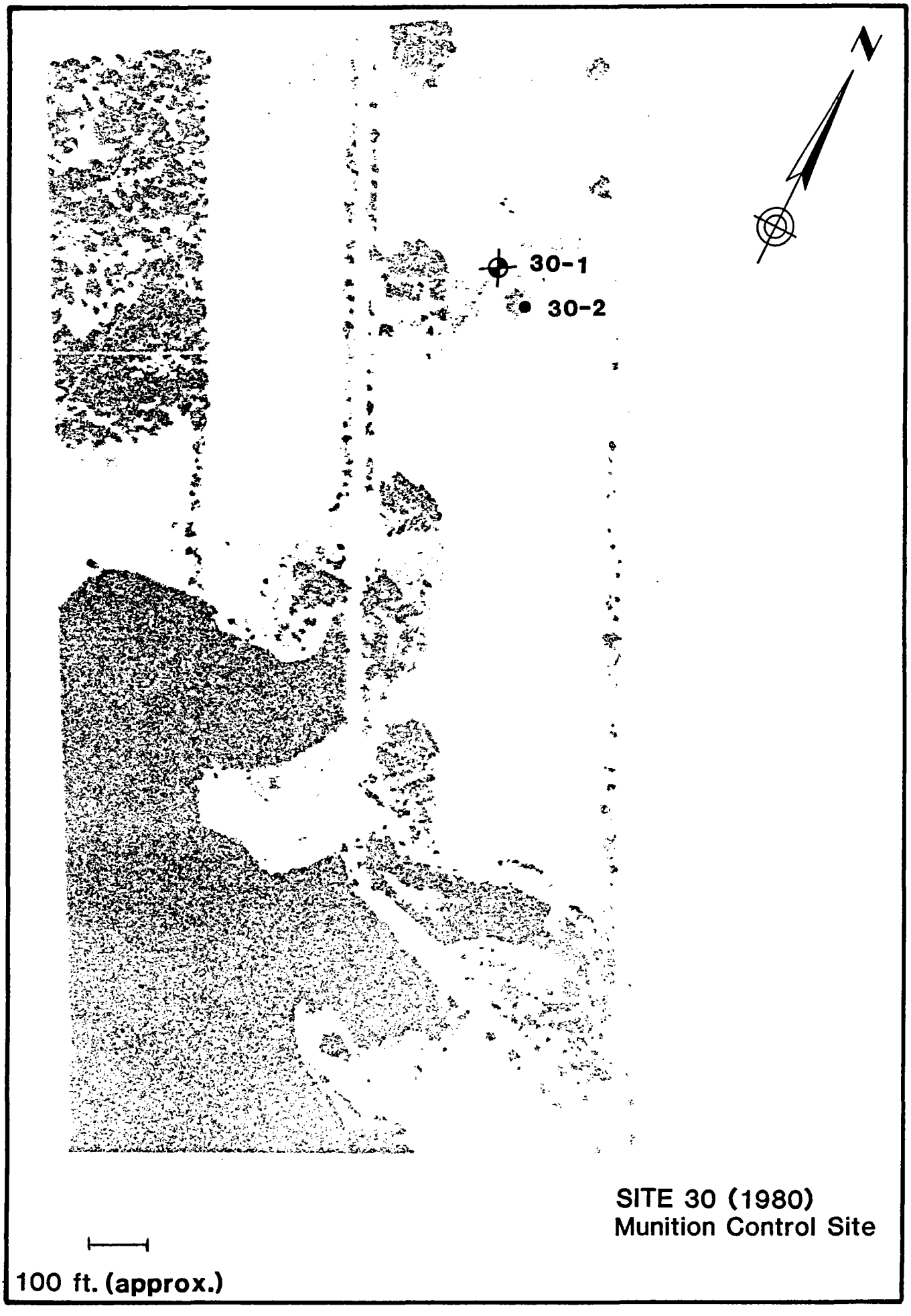
SITE 30

MUNITION CONTROL SITE

A munition control site will be established on an area where the operations involved only ammunitions manufacture.

Sampling and Analysis Scheduled

I.D.	Matrix	Name	Type	Depth	Analysis Set
30-1	Soil	Munition Control	Single Sampling	Surface	D
30-2	Water	Munition Control	Single Sampling	Bailer	A



SITE 30 (1980)
Munition Control Site

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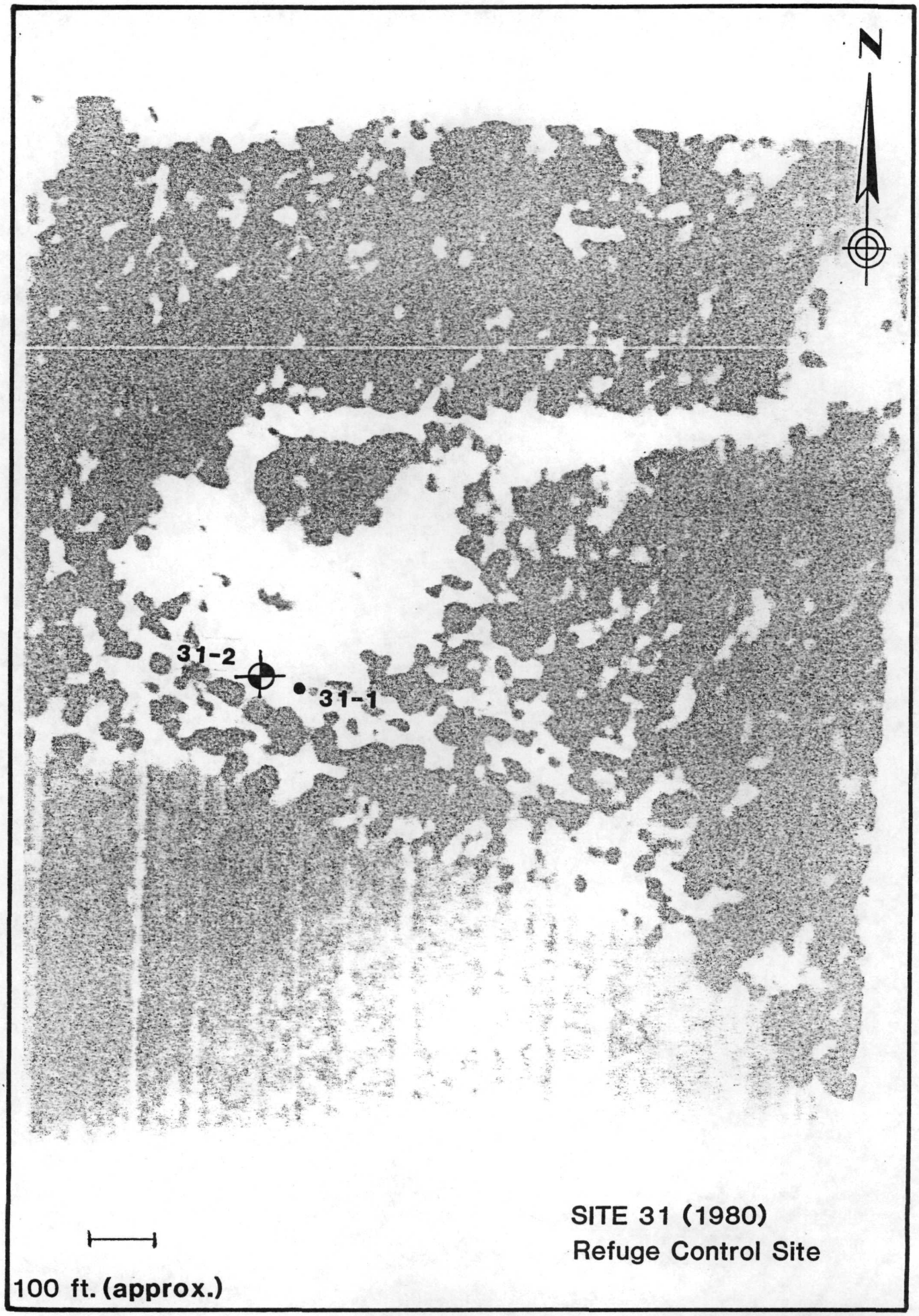
SITE 31

REFUGE CONTROL SITE

A control sampling station will be established on an uncontaminated area of the refuge. Selection of the control site will be coordinated with the Refuge Manager and the QA/QC advisors. During a site visit to the refuge, an area behind the new refuge headquarters was selected as a control site.

I.D.	Matrix	Name	Type	Depth	Analysis Set
31-1	Soil	Refuge Control	Single Sampling	Surface	D
31-2	Water	Refuge Control	Single Sampling	Bailer	A



SITE 31 (1980)
Refuge Control Site

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SITE 32

AREA 9 LANDFILL

Background

The Area 9 Landfill was used during the 1950's and early sixties and was probably closed in 1964. The Landfill is located below approximately 100 yds south of Crab Orchard Lake and approximately 100 yards east of the building complex. Runoff can drain from the landfill into an intermittent creek and then to the Lake. The limits of the landfill are discernable by changes in the topography and vegetation. It is approximately 2.5 acres with a fill thickness of 8 to 10 feet in the middle and 6 feet at the edges. Waste materials are exposed at locations where cover material has eroded. Some areas are void of vegetation.

The volume of the landfill is estimated to be from 16,000 to 35,000 cubic yards. Materials visible on the surface appear to be electrical components consisting of small capacitors, capacitor parts, large chunks of a golden resin, and a large number of 3-inch steel cuplike pieces.

Wastes were burned, compacted in a swale and covered when the landfill was active. Specific compounds of concern include lead, acetate, PCBs (Aroclor 1254 and 1242), and PCB burning products. Other possible materials from capacitor manufacturing include mica, silver, cyanide, aluminum hydroxide, aluminum oxide, gold, copper, zinc, hydrochloric acid, styrene, nitric acid, phosphoric acid, and borates. Other industrial wastes may include cyanides, printing inks and lead-based explosives. A magnetometer survey indicated a high concentration of metals on the east side of the landfill.

Sampling and Analysis Schedule

The sampling program for the Area 9 Landfill will consist of:

1. Landfill soil cores.
2. Surface soils along transect lines along landfill boundaries.
3. Soil cores along Intermittent Creek.
4. Groundwater Monitoring Wells.

1. Landfill Soil Cores:

The Area 9 landfill has been divided into a grid for the collection of soil samples for contaminant analyses. The grid consists of nine triangular-shaped sampling sites. Soil cores from the surface to a depth of 12 feet will be collected from three borings (marked x or 0) at each site. Sample collection criteria and chemical substances to be analyzed for at each of the nine sites making up the grid, in addition to pH, cation exchange capacity, dioxins and dibenzofurans, and explosives residues, are as follows (soil samples will only be composited within and not between locations):

A. Three, 12-foot deep borings for soil core sample collection will be drilled at each location. One core will be collected from each corner of a 50-ft. triangle at each sampling site.

B. Soil subsamples will be collected from the upper 6-inches of each core from the three locations on each triangle, composited and analyzed for PCBs, dioxins and furans.

Soil subsamples will be collected at the 6-foot depths of each core from the three locations on each triangle, composited and analyzed for PCBs, dioxins and furans.

Soil subsamples will be collected from the 12-foot depth of each core from the three locations on each triangle, composited and analyzed for PCBs, dioxins and furans.

C. Soil subsamples will be collected at 1-foot intervals from the surface to a depth of 12 feet on each core on each triangle, composited within squares and analyzed for priority pollutants, dioxins, furans and explosives residues.

2. Surface Soils Along Transect Lines:

The exact boundaries of the landfill are unknown because contaminants could have washed from elevated portions of the landfill onto the lower surrounding area. To identify the extent of contaminant transport from the landfill to surrounding areas, surface soil subsamples will be collected at 3-foot intervals along each of the six transect lines, (two each on the east, south and west side of the landfill) and analyzed for PCBs. The transect lines will be at least 30 feet apart. Three additional transect lines may be sampled and analyzed, depending on results from the first six.

3. Soils Cores - Intermittent Creek:

Six soil cores will be collected from the creek east of the Area 9 landfill. Cores will be collected from the surface to a

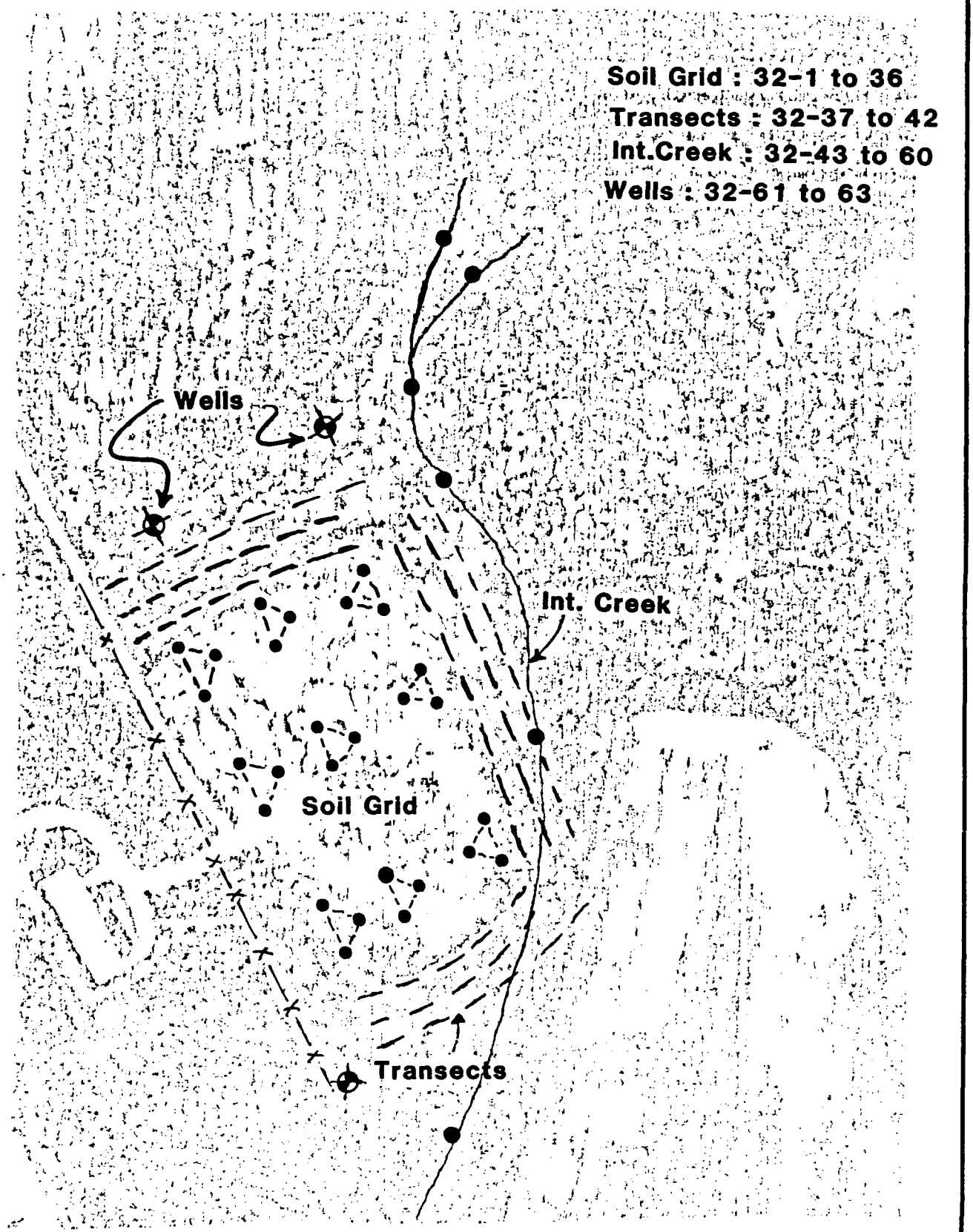
depth of six feet. Individual soil subsamples will be collected from the surface, 3-foot depths and 6-foot depths of each core and analyzed separately for priority pollutants and/or explosives residues detected above background levels in Area 9 landfill soils.

4. Groundwater Monitoring Wells:

There are three existing groundwater sampling wells in the vicinity of the Area 9 Landfill. Duplicate four-liter groundwater samples will be collected at each well by pumping water directly into labeled acid-cleaned jars after the wells have been flushed. These water samples will be analyzed following EPA approved procedures for priority pollutants, explosives residues and the tetra through octa series of dioxins and debenzofurans. The results of these analyses will be reviewed by the U.S. Fish and Wildlife Service. Additional groundwater sampling wells may be drilled or additional analyses performed if justified by chemical substances detected in the initial analyses or for hydrologic reasons.

I.D.	Matrix	Name	Type	Depth	Analysis Set
32-1	Soil	Soil Grid 1	Core Composite at 1' depths	0-12 ft.	D
32-2	Soil	Soil Grid 1-0	Top Composite	0-6 in.	C
32-3	Soil	Soil Grid 1-1	Middle Composite	6-6.5 ft.	C
32-4	Soil	Soil Grid 1-2	Bottom Composite	11.5-12 ft.	C
32-5	Soil	Soil Grid 2	Core Composite at 1' Depths	0-12 ft.	D
32-6	Soil	Soil Grid 2-0	Top Core Composite	0-6 in.	C
32-7	Soil	Soil Grid 2-1	Middle Composite	6-6.5 ft.	C
32-8	Soil	Soil Grid 2-2	Bottom Composite	11.5-12 ft.	C
32-9	Soil	Soil Grid 2-3	Composite at 1' depths	0-12 ft.	D
32-10	Soil	Soil Grid 3-0	Top Core Composite	0-6 in.	C
32-11	Soil	Soil Grid 3-1	Middle Core Composite	6-6.5 ft.	C
32-12	Soil	Soil Grid 3-2	Bottom Core Composite	11.5-12 ft.	C

I.D.	Matrix	Name	Type	Depth	Analysis Set
32-13	Soil	Soil Grid 4	Composite at 1' depths	0-12 ft.	D
32-14	Soil	Soil Grid 4-0	Top Core Composite	0-6 in.	C
32-15	Soil	Soil Grid 4-1	Middle Core Composite	6-6.5 ft.	C
32-16	Soil	Soil Grid 4-2	Bottom Core Composite	11.5-12 ft.	C
32-17	Soil	Soil Grid 5	Composite at 1' depths	0-12 ft.	D
32-18	Soil	Soil Grid 5-0	Top Core Composite	0-6 in.	C
32-19	Soil	Soil Grid 5-1	Middle Core Composite	6-6.5 ft.	C
32-20	Soil	Soil Grid 5-2	Bottom Core Composite	11.5-12 ft.	C
32-21	Soil	Soil Grid 6	Composite at 1' depths	0-12 ft.	D
32-22	Soil	Soil Grid 6-0	Top Core Composite	0-6 in.	C
32-23	Soil	Soil Grid 6-1	Middle Core Composite	6-6.5 ft.	C
32-24	Soil	Soil Grid 6-2	Bottom Core Composite	11.5-12 ft.	C
32-25	Soil	Soil Grid 7-0	Composite at 1' depths	0-12 ft.	D
32-26	Soil	Soil Grid 7-1	Top Core Composite	0-6 in.	C
32-27	Soil	Soil Grid 7-1	Middle Core Composite	6-6.5 ft.	C
32-28	Soil	Soil Grid 7-2	Bottom Core Composite	11.5-12 ft.	C
32-29	Soil	Soil Grid 8	Composite at 1' depths	0-12 ft.	D
32-30	Soil	Soil Grid 8-0	Top Core Composite	0-6 in.	C
32-31	Soil	Soil Grid 8-1	Middle Core Composite	6-6.5 ft.	C
32-32	Soil	Soil Grid 8-2	Bottom Core Composite	11.5-12 ft.	C
32-33	Soil	Soil Grid 9	Composite at 1' depths	0-12 ft.	A
32-34	Soil	Soil Grid 9-0	Top Core Composite	0-6 in.	C
32-35	Soil	Soil Grid 9-1	Middle Core Composite	6-6.5 ft.	C
32-36	Soil	Soil Grid 9-2	Bottom Core Composite	11.5-12 ft.	C
32-37	Soil	W. Transect 1	Composite at 3' Intervals	Surface	B
32-38	Soil	W. Transect 2	Composite at 3' Intervals	Surface	B
32-39	Soil	E. Transect 1	Composite at 3' Intervals	Surface	B
32-40	Soil	E. Transect 2	Composite at 3' Intervals	Surface	B
32-41	Soil	S. Transect 1	Composite at 3' Intervals	Surface	B
32-42	Soil	S. Transect 2	Composite at 3' Intervals	Surface	B
32-43	Sed.	Int. Creek 1-0	Grab	Surface	A
32-44	Sed.	Int. Creek 1-1	Grab	3-foot	A
32-45	Sed.	Int. Creek 1-3	Grab	6-foot	A
32-46	Sed.	Int. Creek 2-0	Grab	Surface	A
32-47	Sed.	Int. Creek 2-1	Grab	3-foot	A
32-48	Sed.	Int. Creek 2-2	Grab	6-foot	A
32-49	Sed.	Int. Creek 3-0	Grab	Surface	A
32-50	Sed.	Int. Creek 3-1	Grab	3-foot	A
32-51	Sed.	Int. Creek 3-2	Grab	6-foot	A
32-52	Sed.	Int. Creek 4-0	Grab	Surface	A
32-53	Sed.	Int. Creek 4-1	Grab	3-foot	A
32-54	Sed.	Int. Creek 4-2	Grab	6-foot	A
32-55	Sed.	Int. Creek 5-0	Grab	Surface	A
32-56	Sed.	Int. Creek 5-1	Grab	3-foot	A
32-57	Sed.	Int. Creek 5-2	Grab	6-foot	A
32-58	Sed.	Int. Creek 6-0	Grab	Surface	D
32-59	Sed.	Int. Creek 6-1	Grab	3-foot	D
32-60	Sed.	Int. Creek 6-2	Grab	6-foot	D
32-61	Water	Well 1	Single Sampling	Bailer	A
32-62	Water	Well 2	Single Sampling	Bailer	A
32-63	Water	Well 3	Single Sampling	Bailer	A



SITE 32 (1960) Area 9 Landfill

100 ft. (approx.)

O'BRIEN&GERE ENGINEERS, INC.

SITE 33

AREA 9 BUILDING COMPLEX

Background

The Area 9 Building Complex was leased during the period from 1946 to 1962 as the Ordill Facility containing the Sangamo Capacitor Division. Manufacturing operations began in the early 1950's. This division manufactured power factor capacitors, AC motor run capacitors, and a variety of DC capacitors. The components were of various types and included aluminum, electrolytes, mica, and silver and lead foil. The Division also manufactured small transformers that used mineral oil as a dielectric.

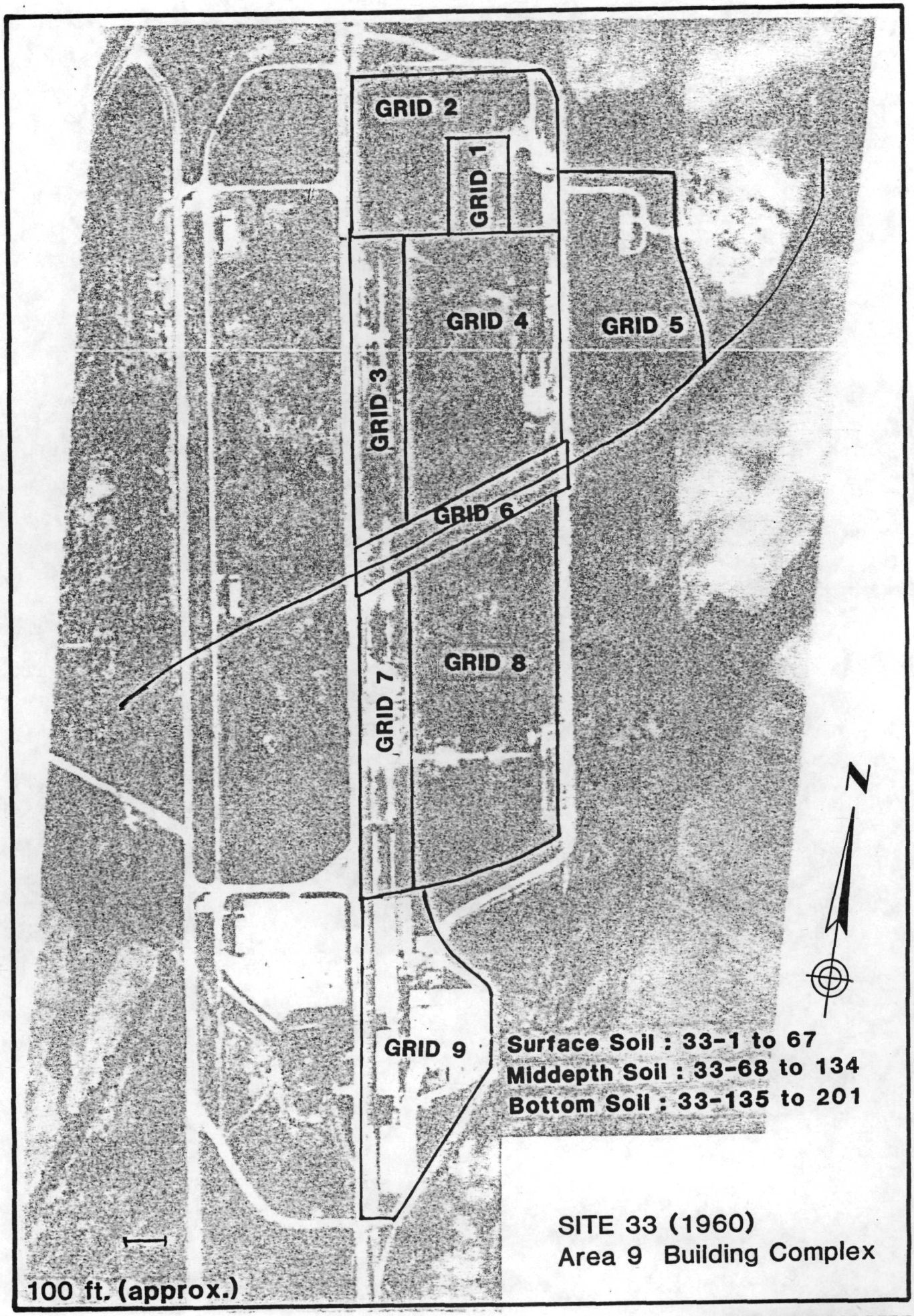
Subsequently, Olin Corporation started using the industrial facilities at the site. Olin manufactured explosives that were used to start jet engines. The company used nitro-glycerine in its operation.

Sampling and Analysis Sequence

Sampling sites will be selected within nine separage grids on the plant property (see attached photo). Sampling locations will emphasize areas of drainage pathways, proximity to buildings, and transportation routes during solid waste disposal. In addition, the sampling locations will be developed using analytical data previously obtained for this site. The surface soils of each core will be first analyzed for PCBs. If PCBs are detected in surface soils of a core, analyses will be performed on soil samples from the mid-depth and the bottom of the core.

Recent results from Olin indicate that PCBs may be present in the soils south or southeast of the buildings in Grid 9. Therefore, the soils from this area also will be sampled. The road ditches leading between the Area 9 buildings and the landfill will be sampled as will any areas that receive drainage from the ditches.

I.D.	Matrix	Name	Type	Depth	Analysis Set
33-1 to 67	Soil	Soil Core 1 to 67	Core Surface	0-6 in.	B
33-68 to 154	Soil	Soil Core 1 to 67	Core Mid-Depth	2-2.5 ft.	B
33-135 to 201	Soil	Soil Core 1 to 67	Core Bottom	3.5-4 ft.	B



SITE 33 (1960)
Area 9 Building Complex

O'BRIEN & GERE
ENGINEERS, INC.

SITE 34

CRAB ORCHARD LAKE

Background

Crab Orchard Lake (completed in 1940) has a surface area of 6,965 acres, a maximum depth of 30 feet, and 635 acre-feet of storage capacity. The watershed drainage area is 109,261 acres. The lake has a retention time of approximately 0.8 years. Water enters the lake through several creeks, including Crab Orchard Creek on the eastern end of the lake and an intermittent creek adjacent to the Area-9 Landfill. Water leaves the lake through Crab Orchard Creek on the western end of the lake. In addition, 280,000 gallons/day of water is used by the Refuge.

The eastern section of the lake is near several manufacturing operations established since the 1940s.

Sampling and Analysis Schedule

Sediment, water, fish, turtles and crayfish will be collected from the lake as follows. The parameters for analysis will be selected on the basis of parameters identified at the study sites on the Refuge.

1. Sediment

   Sediments will be collected from nine sites on Crab Orchard Lake.

   Samples will be collected using an acetone rinsed dredge or, in shallow water, by scooping the sediments directly into the containers. Sediment samples will be stored in labeled, acid-cleaned jars. A sample will consist of a one-liter jar and three 40 ml septum vials of sediment collected from the same

location. The samples in the septum vials will be analyzed for volatile organics. The one-liter sample will be analyzed for priority pollutants other than volatile organics.

2. Water

A complete analysis of water cannot be performed during Phase I because water is to be analysed for contaminants detected in terrestrial sites. It is best to collect fresh water samples for analysis after the analytical parameters have been identified. Therefore, instead of collecting water samples and storing them, water samples will be collected for analysis during Phase II.

Water samples will be collected from the same locations as sediment samples for the Phase II sampling and analysis. However, five water samples will be analyzed during Phase I for drinking water quality parameters and PCBs.

3. Fish

Fish samples will be collected from the lake sites with gill nets or an electroshocker by FWS personnel. Lake fish will be collected during Phase I from the locations shown in the following Figure. These contaminant levels in fish will be compared with Illinois Department of Public Health standards.

Carp and largemouth bass will be collected at each lake and control station. If these species are not available at a station, a species that is available may be substituted. The largest fish collected will be used for analyses. Fish will be analyzed as composite samples. A composite sample will consist of five fish of the same species. One composite sample of each species will be

collected at each station in addition to one duplicate of one species at each station. The composited samples will be analyzed on a whole-fish basis. The control site will be located in the western end of the lake.

4. Crayfish

Crayfish will be collected in minnow traps baited with chicken parts by FWS personnel. Crayfish will be collected from the fish sampling locations. Crayfish may not be present in the deeper water of the mid-lake fish sampling stations. In this case, crayfish will be captured from the shoreline closest to the fish sampling area. A composite crayfish sample will weigh 300 grams or more. Crayfish will be placed in labeled Whirl-pak bags or wrapped in aluminum foil and placed in food-quality plastic bags and frozen before being shipped to the analytical laboratory. Crayfish will be analyzed whole-body.

5. Turtles

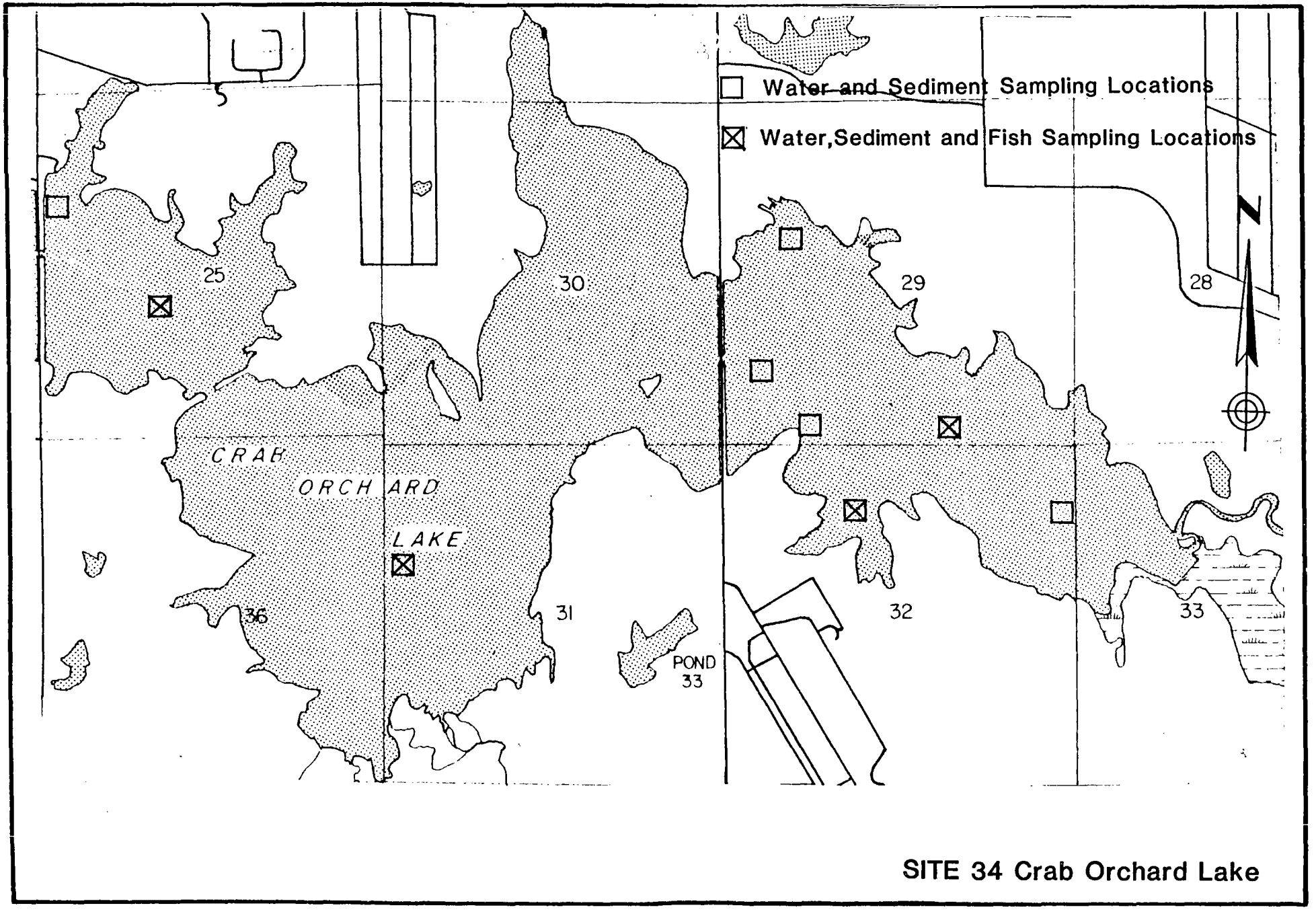
Snapping turtles will be collected from Crab Orchard Lake using liver baited treble hooks on a trot line by FWS personnel. Two or more turtles will be collected from each of the fish sampling locations. It may not be possible to collect the desired number of turtles from all locations. Turtles will be labeled and frozen whole before being shipped to the analytical laboratory.

Turtle livers and fat will be removed under contaminant-free conditions at the laboratory and analyzed separately using EPA approved procedures.

On the following schedule, water samples 34-1 through 34-5 will be collected and analyzed during Phase I. All remaining samples will be analyzed as a part of Phase II.

I.D.	Matrix	Name	Type	Depth	Analysis Set
34-1	Water	Refuge Intake	Grab	NA	E
34-2	Water	Marion Intake	Grab	NA	E
34-3	Water	Marion Res.-Intake	Grab	NA	E
34-4	Water	Refuge Finished Water	Grab	NA	E
34-5	Water	Marion Finished Water	Grab	NA	E
34-6 to 15	Water	Lake 1 to 10	Composite of 3 depths	Surface to 0.8 depth**	A*
34-16 to 20	Sediment	Lake 1 to 10	Grab	Dredge	A*
34-21 to 35	Fish	Lake 1 to 5	Composite of Samples	N/A	A*
34-36 to 38	Turtles	Lake 1 to 3	Single Sampling	Bottom	A*
34-39 to 41	Crayfish	Lake 1 to 3	Composite of 300 gms	Surface	A*

*Only those parameters found at concentrations of significance at any other site.
**Water samples will be collected from the surface, 80 percent of maximum depth and a point midway between these; these samples will be composited.



SITE 34 Crab Orchard Lake

O'BRIEN&GERE ENGINEERS INC

ATTACHMENT 2

PROCEDURES FOR SAMPLING AND SAMPLE PRESERVATION OF WATER AND WASTEWATER

TABLE OF CONTENTS

Introduction

Environmental measurements are required to determine the quality of ambient waters and the character of waste effluents. Standardized analytical methods have been developed to measure the presence and concentration of physical and chemical pollutants in water, wastewater, bottom sediments and solid waste. In addition, techniques to evaluate methods for the concentration, recovery and identification of viruses, bacteria and other microbiological organisms in water and natural waters are also available.

The four basic factors which affect the quality of environmental data are 1) sample collection, 2) sample preservation, 3) analyses, and 4) recording of relevant data and information. If samples are not representative of their original environment, or if the integrity and/or constituents of the samples change between time of sampling and analysis, the control procedures become academic.

This manual was developed to provide general and specific guidelines in sample collection and preservation to help alleviate these problems. Procedures have been standardized as much as possible throughout the manual. Sample preservation methods and holding times are included for the parameters listed for the National Pollutant Discharge Elimination System and Primary Drinking Water Regulations. Special handling or sampling techniques are also included for the individual constituents.

In summary, this manual provides the basis for guidelines in 1) general sampling techniques, 2) preservation of samples for physical, chemical and microbiological analyses and 3) procedures for sampling

water, municipal and industrial wastewaters, surface waters and sludges. The guidelines are procedures specified by enforcement, compliance monitoring or program offices of U.S. Environment Protection Agency.

CHAPTER 1

GENERAL CONSIDERATIONS FOR A SAMPLING PROGRAM

Most definitions of water quality are use-related. Each user produces wastewaters containing pollutants which impacts the environment in different ways. The broad spectrum of ground water, surface waters, lakes, estuaries, coastal waters, municipal wastes, industrial wastewaters and surface run-offs make monitoring of water quality a formidable task. Sampling is the first key element in a monitoring program that must be performed properly to assure valid data. No single sampling program can apply to all types of waters, nevertheless, each sampling program must consider:

1. Objectives of Sampling Program
2. Location of Sampling Points
3. Types of Samples
4. Sample Collection Methods
5. Flow Measurements
6. Field Procedures

OBJECTIVES OF SAMPLING PROGRAMS

There are four major reasons for sampling and analyses program; planning, research or design, process control, and regulation. These objectives in an overall water quality program are interrelated and cover different stages from planning to enforcement.

Most sampling surveys and subsequent analyses are performed to meet the requirements of federal, state, or local regulations. An

example of regulatory monitoring is the National Pollutant Discharge Elimination System (NPDES) established in accordance with the Federal Water Pollution Control Act Amendments of 1977 and 1978 (P.L. 92-500). Specific objectives in collecting regulatory data vary considerably and often overlap, but generally are performed to:

1. Verify self-monitoring data,
2. Verify compliance with NPDES permit,
3. Support enforcement action,
4. Support permit reissuance and/or revision, or
5. Support other program elements such as water quality standards requiring wastewater data.

Usually, the sampling program objectives define the approximate locations for sampling, for example, influent and effluent to a treatment plant or water supply intake. Often, however, the sampling program objectives give only a general indication, such as the effect of a surface runoff on a river quality when assessing the quality of drinking water supplies for a community.

Since the water quality varies from place to place in most water systems, locations appropriate to the information needs of a particular program must be selected. The nature and extent of spatial heterogeneity can vary with time, and can also differ markedly between systems of the same type.

The selected sampling locations must be representative sites. The term "representative point" is defined in 40 CFR, Part 35, subpart B, Appendix A, p. 224, 1976 as a location in surface waters or ground waters at which specific conditions or parameters may be measured in such a manner as to characterize or approximate the quality or

condition of the water body; or a location in process waters or wastewaters where specific conditions or parameters are measured that adequately reflect the actual conditions of those waters or wastewaters.

Factors influencing the selection of sampling locations are:

1. homogenity of the water or wastewater. Turbulence and good mixing resulting from a hydraulic pump and spring and fall turnovers of a lake, respectively, enhance the homogenity or the uniform distribution of the constituents in the body of water.
2. Non-homogenity of the water or wastewater. Poor mixing, for example, stratification in lakes or a river downstream of a waste discharge. Different densities of the constituents, such as floating oils or settling suspended solids. Chemical or biological reactions, such as growth of algae in upper layers of the body of water, causing changes in pH.
3. Other considerations such as pronounced degradation of water quality in specific areas, suitability for flow measurements, convenience and accessability.

The selection of the location of sampling must consider:

1. Homogenity of water or wastewater:
   a. At significant outlets and inputs of lakes, impoundments, estuaries or coastal areas that exhibit eutrophic characteristics.
   b. At locations upstream and downstream of major population and/or industrial centers which have significant discharges into a flowing stream.

c. Upstream and downstream of representative land use areas and morphologic zones.

d. From several locations to obtain the required information.

2. General characteristics of water or wastewater:

a. At representative sites in mainstream of rivers, estuaries, coastal areas, lakes or impoundments.

b. In major water use areas, such as public water supply intakes, commercial fishing areas and recreational areas.

c. At representative sites in the individual waste streams.

d. At the mouths of major or significant tributaries to mainstreams, estuaries or coastal areas.

3. Pronounced water quality degradation:

a. At critical locations (which have the potential for displaying the most pronounced water quality or biological problems) in water quality limiting areas.

b. At critical locations within eutrophic or potentially eutrophic lakes, impoundments, estuaries, or coastal areas.

4. Flow Measurement, i.e., locations where corresponding discharges are known or can be estimated.

5. Convenience, accessibility and practicability are certainly important but they must be secondary to representatives of sampling.

Samples can be collected manually or with automatic samplers. Whichever technique is adopted, the success of sampling program is directly related to the care exercised in the sample collection.

TYPE OF SAMPLE

The type of sample collected depends on the variability of flow, variability of water or wastewater quality, the accuracy required and the availability of funds for conducted the sampling and analytical programs.

A grab sample is defined as an individual discrete sample collected over a period of time not exceeding 15 minutes. It can be taken manually, using a pump, scoop, vacuum, or other suitable device. The collection of a grab sample is appropriate when it is desired to:

1. Characterize water quality at a particular time.
2. Provide information about minimum and maximum concentrations.
3. Allow collection of variable sample volume.
4. Corroborate composite samples.
5. Meet a requirement of a discharge permit.

A composite sample is defined as a sample formed by mixing discrete samples taken at periodic points in time or a continuous proportion of the flow. The number of discrete samples which make up the composite depends upon the variability of pollutant concentration and flow. A sequential composite is defined as a series of periodic grab samples each of which is held in an individual container, then composited to cover a longer time period. Choice of composite type is dependent on the program and relative advantages and disadvantages of each composite type.

Use grab samples when:

1. The stream does not flow continuously such as batch dumps.

2. The water or waste characteristics are relatively constant.
3. The parameters to be analyzed are likely to change with storage such as dissolved gases, residual chlorine, soluble sulfide, oil and grease, microbiological parameters, organics, and pH.
4. Information on maximum, minimum or variability is desired.
5. The history of water quality is to be established based on relatively short time intervals.
6. The spatial parameter variability is to be determined, for example, the parameter variability throughout the cross section and/or depth of a stream or large body of water.

Use composite samples when:

1. Determining average concentrations.
2. Calculating mass/unit time loading.

METHOD OF MANUAL COMPOSITING

When using a constant volume/time proportional compositing method, use previous flow records to determine an appropriate flow volume increment so a representative sample is obtained without exceeding the bottle capacity or supply. The salient features of the method are discussed below.

Suppose a two liter sample is required to be obtained at a constant flow rate over a 24-hour period. The amount of sample to be collected every hour is approximately 80 ml. However, the flow rate at a given sampling site may change over the 24-hour period. In some instances, during the hourly sampling the flow rate may be one-half the previous

rate; in other instances it may be twice the rate when sampling commenced. Thus, during the sampling time the volume collected may vary between 160 ml and 40 ml, respectively.

The portion of each discrete sample is based on its relationship to the total flow. For this reason flows are recorded during each collection and are then compared to the total. An example follows:

1. Suppose the flow per day is 1 MGD = 695 gpm. If the instantaneous flow at sample collection time is 450 gpm, then (695/450) x 80 ml for composite = 123 ml of discrete sample.
2. At next sample collection if the flow is 695 gpm, then place 80 ml of discrete sample into composite.
3. Finally, if the flow is 1000 gpm, then add 56 ml of discrete sample into composite.

For more details, refer to the U.S EPA Handbook for Sampling and Sample Preservation of Water and Wastewater, September, 1982 (EPA-600/4-820-029).

FIELD PROCEDURES

The heart of the sampling program is field operations. If proper precautions and care are not exercised in the field procedures, the entire sampling program will become meaningless despite adequate planning, analytical facilities, and personnel. The key to the success of a field sampling program lies in good housekeeping, collection of representative samples, proper handling and preservation of samples, and appropriate chain of custody procedures.

Good Housekeeping

1. Compose written instructions on field sampling procedures.
2. Prior to use, check sampling equipment to insure good operating conditions and cleanliness. Keep the equipment ready to be used. After the sampling has been completed, clean the equipment and keep it in neat environments.
3. Check primary (e.g. flume) and secondary (e.g. Recorder/transmitter) devices for the following:
   a. Locations
      . At the appropriate place as defined in sampling program.
      . Upstream and downstream conditions meet the requirement of specific installation of primary and secondary devices.
   b. Dimensions of primary devices such as flumes, weirs, and still wells to be sure they are within tolerance limits.
   c. General conditions of channel, primary and secondary devices and stilling wells. Note any unusual wear, debris in channel or distortion of chart paper.
   d. Calibration of primary and secondary devices before actual measurements of flow are taken.
4. Check all sample bottles to avoid contamination. Clean the bottles and if this cannot be done, do not collect the sample.
5. In the laboratory, clean the sample intake tubing by flushing with hot water and then rinsing with distilled water. In the field, rinse several times with sample water.

6. Maintain record of breakdowns in the sampling operations, the problems encountered with different equipment and how they were resolved. This information indicates the reliability of the equipment, the problem areas that need to be brought to the manufacturer's attention, and considerations for future procurements.
7. Hold training sessions for field sampling teams.

Guidelines for Representative Sample

To obtain representative samples, follow these guidelines:

1. Collect the sample where water is well mixed, that is near a Parshall flume or at a point of hydraulic turbulence such as downstream of a hydraulic jump. Certain types of weirs and flumes tend to enhance the settling of solids upstream and accumulate floating solids and oil downstream, therefore such locations should be avoided as a sample source. For low level turbulence, mechanical or air mixing should be used to induce turbulence except when dissolved gases or volatile materials are being sampled.
2. Collect the sample in the center of the channel at 0.4 to 0.6 depth from the bottom where the velocity of flow is average or higher than average and chances of solids settling is minimum. This depth avoids bottom bed loads and top floating materials such as oils and grease.
3. In a wide channel, divide the channel cross section into different vertical sections so that each section is equal width. Take a representative sample in each vertical section.

4. In a deep stream or lake, collect the sample at different depths. In those cases of wide and deep streams the samples can be composited or analyzed individually depending upon the program objective.
5. When manual sampling with jars, place the mouth of the collecting container below the water surface and facing flow to avoid an excess of floating material. Keep the hand away from the mouth of the jar as far as possible.
6. Additional guidelines for manual sampling:
   a. Sample facing upstream to avoid contamination.
   b. Force sampling vessel through the entire cross section of the stream wherever possible.
   c. Drop an inverted bucket and jerk line just before impact with the water surface.
   d. Be certain that the sampler closes and opens at the proper time when sampling with a depth integrating sampler; with a point sampler, be certain that sampler opens at a proper depth. If a doubt exists, discard the sample and re-sample.
7. When sampling, it is necessary to fill the bottles completely if the samples are to be analyzed for volatile organics, O_2, CO_2, NH_3, H_2S, free chlorine, pH, hardness, SO_2, NH^+_4, FE^{++}, oil and grease, acidity or alkalinity. When sampling for bacteria or suspended solids, it is necessary to leave an airspace in the sample container to allow mixing before subsampling.
8. Collect sufficient volume to allow duplicate analyses and quality assurance testing (split or spiked samples). The required

sample volume is a summation of that required for each parameter of interest. Refer to USEPA's Methods for Chemical Analyses for Water and Wastewater, 1979, EPA 600/4-79-0202 for the volume required for analysis of a specific parameter, or the laboratory director for minimum volumes to be collected.

9. Maintain an up-to-date log book which notes possible interferences, environmental conditions and problem areas.

10. Since mathematical relationship between volumetric flow and height (or depth) of flow is nonlinear, composite flow proportional samples in relation to the total volume of flow as proposed to gauge height or raw measurement of a secondary device.

11. If samples are taken from a closed conduit via a valve or faucet arrangement, allow sufficient flushing time to insure that the sample is representative of the supply, taking into account the diameter, length of the pipe to be flushed and the velocity of the flow.

CHAPTER 2

SAMPLE PRESERVATION

Complete preservation of samples, either domestic sewage, industrial wastes, or natural waters, is a practical impossibility. Regardless of the nature of the sample, complete stability for every constituent can never be achieved. At best, preservation techniques can only retard the chemical and biological changes that take place in a sample after the sample is removed from the parent source. To maintain the integrity of the sample, appropriate selection of containers, pretreatment of containers if necessary and the holding times form the integral part of the sample preservation program. Preservation guidelines for NPDES samples are given in Table I below. Recommendations for sampling volume are given in Table II.

TABLE I

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter	Container[1]	Preservative[2, 12]	Maximum Holding Time[3]
Bacterial Tests			
Coliform, fecal and total	P,G	Cool, 4°C 0.008% $Na_2S_2O_3$[6]	6 hours
Fecal streptococci	P,G	Cool, 4°C 0.008%, $Na_2S_2O_3$[6]	6 hours
Inorganic Tests			
Acidity	P,G	Cool, 4°C	14 days
Alkalinity	P,G	Cool, 4°C	14 days

(Table I Continued)

Parameter	Container[1]	Preservative[2, 12]	Maximum Holding Time[3]
Ammonia	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 days
Biochemical oxygen demand	P,G	Cool, 4°C	48 hours
Biochemical oxygen demand, carbonaceous	P,G	Cool, 4°C	48 hours
Bromide	P,G	None required	28 days
Chemical oxygen demand	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 days
Chloride	P,G	None required	28 days
Chlorine, total residual	P,G	None required	Analyze immediately
Color	P,G	Cool, 4°C	48 hours
Cyanide, total and amenable to chlorination	P,G	Cool, 4°C NaOH to pH > 12 0.6g ascorbic acid[6]	14 days[9]
Fluoride	P	None required	28 days
Hardness	P,G	HNO_3 to pH < 2	6 months
Hydrogen ion (pH)	P,G	None required	Analyze immediately
Kjeldahl and organic Nitrogen	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 days
Metals[4]			
Chromium VI	P,G	Cool, 4°C	24 hours
Mercury	P,G	HNO_3 to pH < 2	28 days
Metals, except above	P,G	HNO_3 to pH < 2	6 months
Nitrate	P,G	Cool, 4°C	48 hours

(Table I Continued)

Parameter	Container[1]	Preservative[2, 12]	Maximum Holding Time[3]
Nitrate-nitrite	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 days
Nitrite	P,G	Cool, 4°C	48 hours
Oil and grease	G	Cool, 4°C H_2SO_4 to pH < 2	28 days
Organic carbon	P,G	Cool, 4°C HCl or H_2SO_4 to pH < 2	28 days
Orthophosphate	P,G	Filter immediately Cool, 4°C	48 hours
Oxygen, Dissolved Probe	G bottle and top	None required	Analyze immediately
Winkler	G bottle and top	Fix on site and store in dark	8 hours
Phenols	G only	Cool, 4°C H_2SO_4 to pH < 2	28 days
Phosphorus (elemental)	G	Cool, 4°C	48 hours
Phosphorus, total	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 days
Residue, total	P,G	Cool, 4°C	7 days
Residue, Filterable	P,G	Cool, 4°C	7 days
Residue, Non-filterable (TSS)	P,G	Cool, 4°C	7 days
Residue, settleable	P,G	Cool, 4°C	48 hours
Residue, volatile	P,G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific conductance	P,G	Cool, 4°C	28 days
Sulfate	P,G	Cool, 4°C	28 days

(Table I Continued)

Parameter	Container[1]	Preservative[2, 12]	Maximum Holding Time[3]
Sulfide	P,G	Cool, 4°C, add zinc acetate plus sodium hydroxide to pH > 9	7 days
Sulfite	P,G	Cool, 4°C	Analyze immediately
Surfactants	P,G	Cool, 4°C	48 hours
Temperature	P,G	None required	Analyze immediately
Turbidity	P,G	Cool, 4°C	48 hours
Organic Tests			
Purgeable halocarbons	G, Teflon-lined septum	Cool, 4°C 0.008% $Na_2S_2O_3$[6]	14 days
Purgeable aromatics	G, Teflon-lined septum	Cool, 4°C 0.008% $Na_2S_2O_3$[6] HCl to pH 2[10]	14 days
Acrolein and acrylonitrile	G, Teflon-lined septum	Cool, 4°C 0.008% $Na_2S_2O_3$[6] Adjust pH to 4-5[11]	14 days
Phenols	G, Teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction
Benzidines	G, Teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction
Phthalate esters	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction

(Table I Continued)

Parameter	Container[1]	Preservative[2, 12]	Maximum Holding Time[3]
Nitrosamines	G, Teflon-lined cap	Cool, 4°C store in dark 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction
PCB's	G, Teflon-lined cap	Cool 4°C[8] pH 5-9	7 days until extraction, 40 days after extraction
Nitroaromatics and isophorone	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
Polynuclear aromatic hydrocarbons	G, Teflon-lined cap	Cool, 4°C store in dark 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction
Haloethers	G, Teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction
Chlorinated hydrocarbons	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
TCDD	G, Teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction
Pesticides Tests			
Pesticides	G, Teflon-lined cap	Cool, 4°C pH 5-9[8]	7 days until extraction, 40 days after extraction
Radiological Tests			
Alpha, beta and radium	P,G	HNO_3 to pH < 2	6 months

NOTES

1. Polyethylene (P) or Glass (G).
2. Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time. Some samples may not be stable for the maximum time period give in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample stability.
4. Samples should be filtered immediately on-site before adding preservative for dissolved metals.
5. Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
6. Should only be used in the presence of residual chlorine.
7. For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

8. The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted with 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.
9. Maximum holding time is 24 hours when sulfide is present.
10. Sample receiving no pH adjustment must be analyzed within seven days of sampling.
11. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
12. When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table I, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

TABLE II

RECOMMENDATIONS FOR SAMPLING VOLUME
OF SAMPLES ACCORDING TO MEASUREMENT

Measurement	Volume	Container
Metals	1 pt	plastic bottle/cap
Phenols	1 qt	glass bottle/teflon lined cap only
Pesticides	1 qt	glass bottle/teflon lined cap
Herbicides	1 qt	glass bottle/teflon lined cap
Inorganics	1 qt	plastic bottle/cap
Cyanide	1 pt	plastic bottle/cap
Nutrients	1 pt	plastic bottle/cap
Demand	1 pt	plastic bottle/cap
VHO	40 ml	duplicate glass bottle/teflon septum cap
THMS	40 ml	duplicate glass bottle/teflon septum cap
Extractable (base/neutrals/ acid) organics	1 qt	glass bottle/teflon lined cap
Solids	1 qt	plastic bottle/cap
Oil & Grease	1/2 gal	glass bottle/teflon lined cap only

CONTAINERS

A variety of factors affect the choice of containers and cap material. These include resistance to breakage, size, weight, interference with constituents, cost and availability. There are also various procedures for cleaning and preparing bottles depending upon the analyses to be performed on the sample.

The two major types of container materials are plastic and glass.

Glass:

1. Kimax Or Pyrex brand - borosilicate
2. Vycor - generally lab ware
3. Ray-Sorber Low-Actinic - generally lab ware
4. Corex - generally lab ware

Plastic:

1. Conventional polyethylene
2. Linear polyethylene
3. Polypropylene
4. Polycarbonate
5. Rigid polyvinyl chloride
6. Teflon

All these materials have various advantages and disadvantages. Kimax or Pyrex brand borosilicate glass is inert to most materials and is recommended where glass containers are used. Conventional polyethylene is to be used when plastic is acceptable because of reasonable cost and less absorption of metal ions. The specific situation will determine the use of glass or plastic. However, use glass containers for pesticides, oil and grease, and other organics.

There are two major types of plastic used in container caps: polyethylene and bakelite with liners. Ploythylene caps are recommended for ease of cleaning unless oil and grease analyses are to be performed. Caps with Teflon liners should be used for pesticides and oil and grease samples. Silicone rubber material should be avoided for Trace Metals because of Zinc contaminations.

The following procedure should be followed to wash containers and caps for inorganic and general parameters:

1. Wash containers and caps with a non-phosphate detergent and scrub strongly with a brush (if possible wash liners and caps separately).
2. Rinse with tap water, then distilled water.
3. Invert to drain and dry.
4. Visually inspect for any contamination prior to storage.
5. If the container requires additional cleaning, rinse with a chromic acid solution (35 mL saturated sodium dichromate solutiuon in 1 liter of sulfuric acid - this solution can be reused). Then rinse with tap water and distilled water and dry as indicated above.

For certain parameters, a special cleaning procedure is needed to avoid adsorption or contamination due to interaction with container walls. These procedures are outlined below:

1. Metals: If metals are to be analyzed, rinse the container with a solution of one part nitric acid to four parts water, then with distilled water. If phosphorus is to be analyzed, rinse the container with a solution of one part hydrochloric acid to one part water, followed by distilled water. Treat the caps similarly.
2. Organics: If Oil and Grease or Pesticides are to be analyzed, rinse the sample container with methylene chloride, followed by acetone. For Pesticide analysis, use pesticide grade hexane or acetone.

3. Sterilization: For microbiological analyses, sterilize the container and its stopper/cap by autoclaving at 121°C for 15 minutes or by dry heat at 180°C for two hours. Heat-sensitive plastic bottles may be sterilized with ethylene oxide at low temperatures. Wrap bottles in kraft paper or cover with aluminum foil before sterilization to protect against contamination. An acceptable alternative for emergency or field use is sterilization of containers by boiling in water for 15 minutes.

CHAPTER 3

SAMPLING FOR TRACE ORGANICS

Analytical procedures for the identification of organic compounds can be found in a number of publications. However, analytical results are only meaningful if the sample analyzed is truly a representative sample of the media you are testing. Chemical analysis for organics present at trace levels places high demands on sampling techniques.

Although continuous automatic sampling is probably the best method for collecting truly representative samples, certain precautions must be taken. Automatic sampling equipment must be free of Tygon and other potential sources of contamination such as plastic, or rubber components. Tygon tubing is a potential source of phthalate ester contamination. Teflon is acceptable and may be used in the sampling system as required.

Automatic samplers can be used to collect composited samples. EPA's 600 series methods for analyzing non-volatile organic priority pollutants reference these types of automatic samplers.

The configuration and materials of a container which can be utilized in the collection and storage of organic containing samples are somewhat varied. However, the following criteria should be met:

1. Non-purgeable samples must be collected in amber glass containers in a liter or quart volume and preferably of French or Boston round design. Various glass vials have also proved to be adequate.

2. Container caps should be treaded to screw onto the container. Caps must be lined with Teflon. Foil may be substituted if sample is not corrosive.
3. Purgeable sample must be collected in 40 mL borosilicate glass vials with screw caps (Pierce #13075 or equivalent). The septa used must be Teflon faced silicon (Pierce #12722 or equivalent).

SAMPLING PROCEDURE AND PRETREATMENT OF SAMPLE EQUIPMENT

Pretreatment of Equipment

The pretreatment technique should be dictated by the analysis to be performed. The general pretreatment technique for sample and storage containers is to:

1. Wash bottles with hot detergent water.
2. Rinse thoroughly with tap water followed by three or more rinses with organic-free water.
3. Rinse with interference free redistilled solvent such as acetone or methylene chloride and dry in contaminant free air at room temperature. Protect from atmospheric or other sources of contamination. Caps and liners for bottles must also be solvent rinsed as above.

If automatic samplers are to be employed, use the peristaltic pump type with a single 8 - 10 liter (2.5 - 3.0 gallons) glass container. Vacuum type automatic samplers can be used if sample containers are glass. The procedure outlined above should be followed for the pretreatment of the containers. In addition all tubing and other parts of

the sampling system must be scrubbed with hot detergent water and thoroughly rinsed with tap water and blank water prior to use. Further rinsing with interference free acetone or methylene chloride is advised when tubing and other parts permit, i.e., are not susceptible to dissolution by the solvent.

Sampling Procedure

Purgeables

Collect grab samples in glass containers. The procedure for filling and sealing sample containers is as follows: Slowly fill each container to overflowing (Figure 1). Carefully set the container on a level

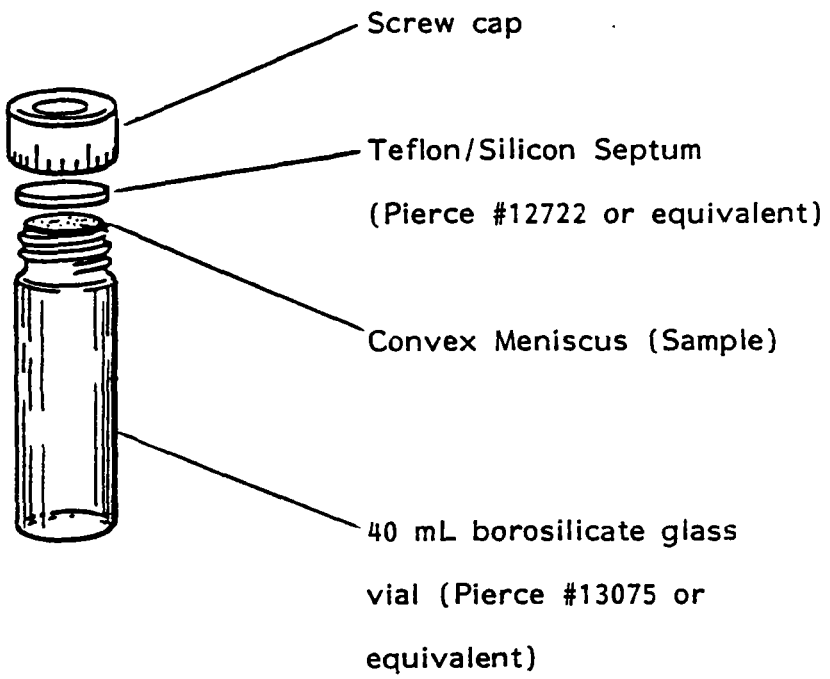


Figure 1. Collection Bottle for Trace Organics

surface. Place the septum Teflon side down on the convex sample meniscus. Seal the sample with the screw cap. To insure that the sample has been properly sealed, invert the sample and lightly tap the lid on a solid surface. The absence of entrapped air bubbles indicates a proper seal. If air bubbles are present, open the bottle, add additional sample, and reseal (in same manner as stated above). The sample must remain hermetically sealed until it is analyzed. Maintain samples at 4°C (39°F) during transport and storage prior to analysis. If the sample is taken from a water tap, turn on the water and permit the system to flush. When the temperature of the water has stabilized, adjust the flow to about 500-mL/minute and collect samples as outlined above.

Non-Purgeables

Collect grab samples in glass containers. Conventional sampling practices should be followed, except that the bottle must not be pre-washed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be free of Tygon and other potential sources of contamination.

CHAPTER 4

SAMPLING MUNICIPAL WASTEWATERS

BACKGROUND

Municipal wastewaters are collected and treated by chemical, physical, and/or biological means prior to discharge to surface waters. Up to three stages, primary, secondary and tertiary, are commonly used at municipal treatment plants. The wastewater characteristics vary with the size and habits of the community, the type of collection system (combined or separate), the amount of infiltration and the volume and type of industrial discharges entering the system.

The NPDES Compliance Sampling Manual indicates that sampling programs must include a minimum of a 24 hours of operating day composite supplemented by two or more grab samples. With highly variable wastewater characteristics or flow rate changes, additional sampling is required. A composite sample is defined as a minimum of eight discrete samples taken, proportional to flow rate, over the compositing period.

LOCATION OF SAMPLING POINTS

Collect the sample at the location(s) specified in the permit. At these locations collect the sample in the center of the channel at 0.4 to 0.6 depth where the flow is turbulent, well mixed, and the settling of solids is minimal. Sampling at 0.4 to 0.6 depth will avoid skimming of the water surface or dragging the channel bottom.

Influent

Influent wastewaters are preferably sampled at points of highly turbulent flow in order to insure good mixing; however, in many instances the desired location is not accessible. In all cases, samples should be collected upstream from recirculated plant supernatant and sludges.

Effluent

Collect effluent samples at the most representative site downstream from all entering waste streams. When manuallly compositing effluent samples according to flow where no flow measuring device exists, use the influent flow measurement without any correction for time lag.

Pond Sampling

Composite samples from ponds with long detention times may not be representative because of the tendency of lagoons to short circuit. If dye studies or past experience indicate a homogeneous discharge, grab samples may be representative of the waste stream.

CHAPTER 5

SAMPLING INDUSTRIAL WASTEWATERS

BACKGROUND

Industrial wastewaters vary significantly in pollution characteristics. This chapter presents general guidelines and considerations so that effective sampling programs can be established for varied situations.

Sampling of industrial wastewaters is required by regulatory agencies for the NPDES permit program. The location or sampling points, frequency and sample type are specified in the NPDES permit. At the time of NPDES permit modifications, incorporate the recommendations of Compliance Sampling Inspections.

Consider variable plant operations when determining frequency:

1. Seasonal operation
2. Less than 24 hours per day operation
3. Special times during the day, week or month set aside for cleanup
4. Any combination of the above

When monitoring these types of operations, it is necessary to sample during normal working shifts in the season of productive operation.

To achieve process control or to design and implement in-plant pollution control programs, selection of proper in-plant sample location is important. Use the following procedures to determine the sampling locations:

1. Become familiar with the plant processes and sources of wastes from unit operations.
2. Ascertain the sewer layout in the plant. If a sewer plan exists thoroughly review the sewer plan and examine each sewer to determine its course and destination. Where a sewer plan is not available, the only practical way to determine the sewer layout is by dye-tracing.
3. Determine the exact source and the point at which each waste stream enters the sewer.
4. Sample each waste stream and plant outfall. By doing so, each waste stream is characterized and the outfall characterizes the total plant effluent.
5. Sample each batch discharge.
6. If a point of upset exists within the plant, establishment of a sampling station or monitoring equipment at that point will allow early detection.
7. If data on different waste streams is available from past records, use statistical techniques as an aid to establish the critical sampling locations within the plant.

TYPE OF SAMPLE

The permit will specify the type of sample, grab or composite, for effluent monitoring, but consider both types for in-plant monitoring. Where in-plant data do not exist, conduct a preliminary survey with production personnel of each unit process to determine the chemical reactions, production variability, location of individual waste streams

and their potential variability, and potential chemical constituents in each waste stream. After careful analysis of the unit process, select the appropriate type of sample to be collected. Collect proportional composite samples to determine the average amount of pollutant or collect grab samples:

1. If a batch discharge is to be characterized.
2. If the flow is homogeneous and continuous with relatively constant waste characteristics so a grab sample is representative of the stream.
3. When the extremes of flow and quality characteristics are needed.
4. When one is sampling for a parameter requiring that the entire sample be used for analysis with no interior transfers of containers, for example, oil and grease.
5. When sampling for parameters which change character rapidly such as dissolved gases or those which cannot be held for a long length of time before analyses, for example, bacteria counts, chlorine, dissolved oxygen and sulfide.

METHOD OF SAMPLING

Choose manual or automatic sampling depending upon which method is best for the specific sampling program. Only trained personnel should be entrusted the task of sample collection.

The volume of sample to be taken is determined by the number of analyses to be performed on the sample. If this has not been determined, a grab sample volume, a minimum of 7.57L (2 gallon) and an

individual composite volume of 100 milliliters (0.21 pints) should be taken. The container type is also contingent upon the analysis to be run.

PRESERVATION AND HANDLING OF SAMPLES

The preservation, holding times, and materials associated with sampling depends upon the parameters to be analyzed. Refer to Chapter 2 for specific instructions.

CHAPTER 6

SAMPLING SURFACE WATERS

BACKGROUND

The sampling of rivers and streams, lakes and aquatic organisms, and their associated bottom sediments are considered in this chapter. The decisions regarding analytical parameters must be made at the beginning of the study in order to develop a rational sampling program.

LOCATION OF SAMPLING POINTS

Select the study site based on the program objectives, the parameters of interest, and the type of sample. For example, the following guidelines are suggested in the EPA Model State Water Monitoring Program for selecting long term biological trend monitoring stations:

1. At key locations in water bodies which are of critical value for sensitive uses such as domestic water supply, recreation, propogation, and maintenance of fish and wildlife.
2. In the main stream upstream and downstream from the confluence of major tributaries and in the tributary upstream from the confluence with the main stream.
3. Near the mouths of major rivers where they enter an estuary.
4. At locations in major water bodies potentially subject to inputs of contaminants from areas of concentrated urban, industrial, or agricultural use.

5. At key locations in water bodies largely unaffected by man's activities.

FREQUENCY AND METHOD OF SAMPLING

While the frequency of sampling will often be determined by the program, use the Model State Water Monitoring Program guidelines for guidance in trend monitoring.

While compositing of individual grab samples is permitted for most chemical parameters, as a rule biological samples are not composited. For biological parameters, collect single grab samples.

VOLUME OF SAMPLE AND CONTAINER TYPE

Refer to Chapter 2 for specific information relative to the chemical parameters which are to be analyzed. In general, do not use metal samplers for trace metal nor use plastics for sampling trace organics.

PRESERVATION HANDLING OF SAMPLES

Refer to Chapter 2 for specific information regarding preservation and handling of samples relative to the chemical parameters to be analyzed, and to the EPA biological methods manual for aquatic organism preservatives.

CHAPTER 7

SAMPLING OF GROUND WATER

BACKGROUND

Ground water accounts for the base flow of all perennial streams, over 90 percent of the world's fresh water resources, and one half the drinking water in the United States, yet has traditionally received only token scientific attention. Although surface and ground waters are inseparable parts of the same hydrologic system with the waters of each flowing alternately between the two components, water resource planners have often considered them as separate entities.

Methods of collecting a representative ground water sample are much more difficult and expensive in this often remote and relatively inaccessible environment. The subsurface is an extremely complex system subject to extensive physical, chemical and biological changes within small vertical and horizontal distances.

OBJECTIVES OF GROUND WATER SAMPLING

Samples from a monitoring well represent a small part of an aquifer horizontally and in many cases, vertically. Unlike its surface counterpart where a sample can be arbitrarily taken at any point in the system, moving a ground water sampling point implies the installation of additional monitoring wells. Because of the difficulty and expense, it is essential that sampling objectives be firmly established well in advance of field activities. These objectives will dictate the parameters to be

measured, the necessary reliability of the water quality data, and analytical methodology and thence the sampling procedures necessary to meet these objectives.

If the objective is simply to determine the presence or absence of a conservative pollutant in a particular water supply, it is simple and relatively inexpensive to collect a sample at a water tap. However, if the organic pollutant or pollutants and predict the eventual fate, then soil cores, monitoring wells and special sampling equipment may increase efforts and cost several orders of magnitude.

The unstable nature of many chemical, physical, and microbial constituents in ground water and subsurface limit the sample collection and analyses options. However, certain factors should be considered when collecting representative samples:

1. Ground water moves slowly, therefore a slow rate of change of water quality parameters.
2. Temperatures are relatively constant in the subsurface, therefore the sample temperature may change significantly when brought to the surface. This change can alter chemical reaction rates, reverse cationic and anionic ion exchanges on solids, and change microbial growth rates.
3. A change in pH can occur due to carbon dioxide adsorption and subsequent changes in alkalinity. Oxidation of some compounds may also occur.
4. Dissolved gases such as hydrogen sulfide may be lost at the surface.

5. Integrity of organic samples may be affected by problems associated with either adsorption or contamination from sampling materials and volatility.
6. Both soils and ground waters may be so severely contaminated as to present a health or safety problem to sampling crews.

COLLECTION OF GROUND WATER SAMPLES

The importance of proper sampling of wells cannot be overemphasized. Even though the well being sampled may be correctly located and constructed, special precautions must be taken to ensure that the sample taken from that well is representative of the ground water at that location and that the sample is neither altered nor contaminated by the sampling and handling procedure.

To obtain a representative sample of the ground water it must be understood that the composition of the water within the well casing and in close proximity to the well is probably not representative of the overall ground water quality at that sampling site. This is due to the possible presence of drilling contaminants near the well and because important environmental conditions such as the oxidation reduction potential may differ drastically near the well from the conditions in the surrounding water bearing materials. For these reasons it is highly desirable that a well be pumped or bailed until the well is thoroughly flushed of standing water and contains fresh water from the aquifer. The recommended length of time required to pump or bail a well before sampling is dependent on many factors including the characteristics of the well, the hydrogeological nature of the aquifer, the type of

sampling equipment being used, and the parameters being sampled. The time required may range from the time needed to pump or bail one bore volume to the time needed to pump several bore volumes. A common procedure is to pump or bail the well until a minimum of four to ten bore volumes have been removed.

Other factors which will influence the time required to flush out a well before sampling include the pumping rate and the placement of the pumping equipment within the column of water in the well bore. Care should be taken to ensure that all of the water within the well bore is exchanged with fresh water. For example, recent studies have shown that if a pump is lowered immediately to the bottom of a well before pumping, it may take some time for the column of water above it to be exchanged if the transmissivity of the aquifer is high and the well screen is at the bottom of the casing. In such cases the pump will be pumping primarily water from the aquifer. Removing all water from the well bore is only possible if the well is pumped dry and alternative approaches have been suggested, i.e.,:

(a) monitor the water level in the well while pumping. When the water level has "stabilized" most if not all of the water being pumped is coming from the aquifer

(b) monitor the temperature and pH of the water while pumping. When these two parameters "stabilize," it is probable that little or no water from casing storage is being pumped.

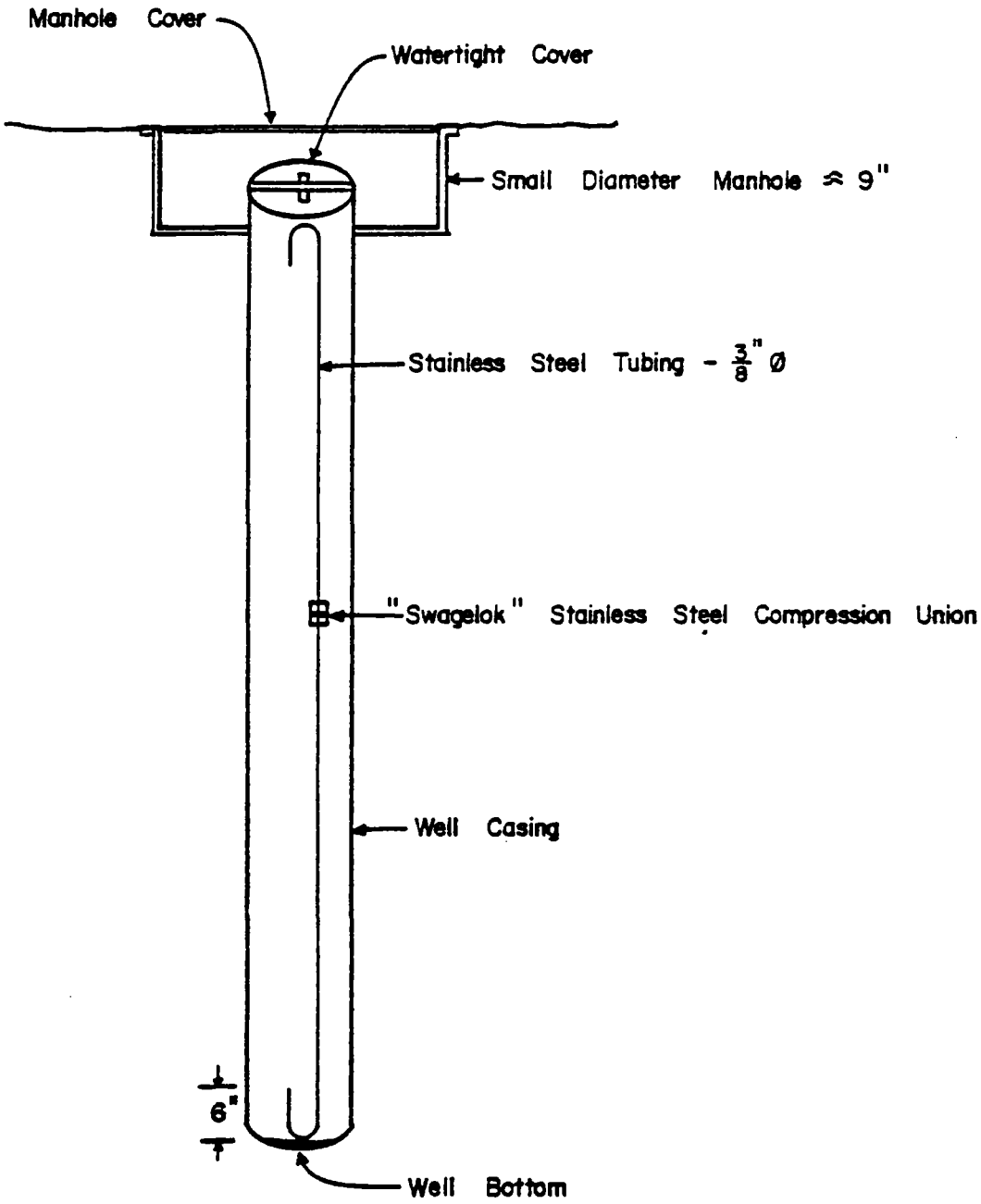
WELL SAMPLING TECHNIQUE

The sampling methods used for wells are controlled by three factors: 1) water elevation (distance between the top of the well casing and the water surface) which is less than approximately 24 feet, 2) water elevation greater than 24 feet, and 3) degree of well contamination.

At each well to be sampled, the first procedure is to unlock and remove the well cap and take the elevation with "Soiltest" water level indicator. In most cases, the water elevations are between 6 and 24 feet, and it is possible to install 3/8" ASTM 304 stainless steel tubing for use in obtaining a sample. The stainless steel tubing is thoroughly cleaned with hexane and rinsed with methanol before installation. Once installed, the tubing is left in the well permanently (see Figure 2).

To sample a clean well water elevation less than 24 feet, the top end of the stainless steel tubing is connected to a 250 ml flask by pushing it through a silicone stopper (see Figure 3). A second piece of stainless steel tubing is pushed through the stopper and connected to a 120v "Jabsco" impeller pump with a piece of heavy-duty vacuum tubing. A third piece of heavy-duty vacuum tubing is connected to the pump discharge and runs to a large 1000 ml vacuum flask which is evacuated by a large capacity manually operated vacuum pump. The vacuum applied to the system draws water up the tubing into the first flask and through the pump which in effect primes the pump. The pump is started and the large vacuum flask is removed. The pump is allowed to run for ten minutes.

STAINLESS STEEL TUBING INSTALLATION



NOT TO SCALE

WELL PUMPING - SAMPLING SYSTEM

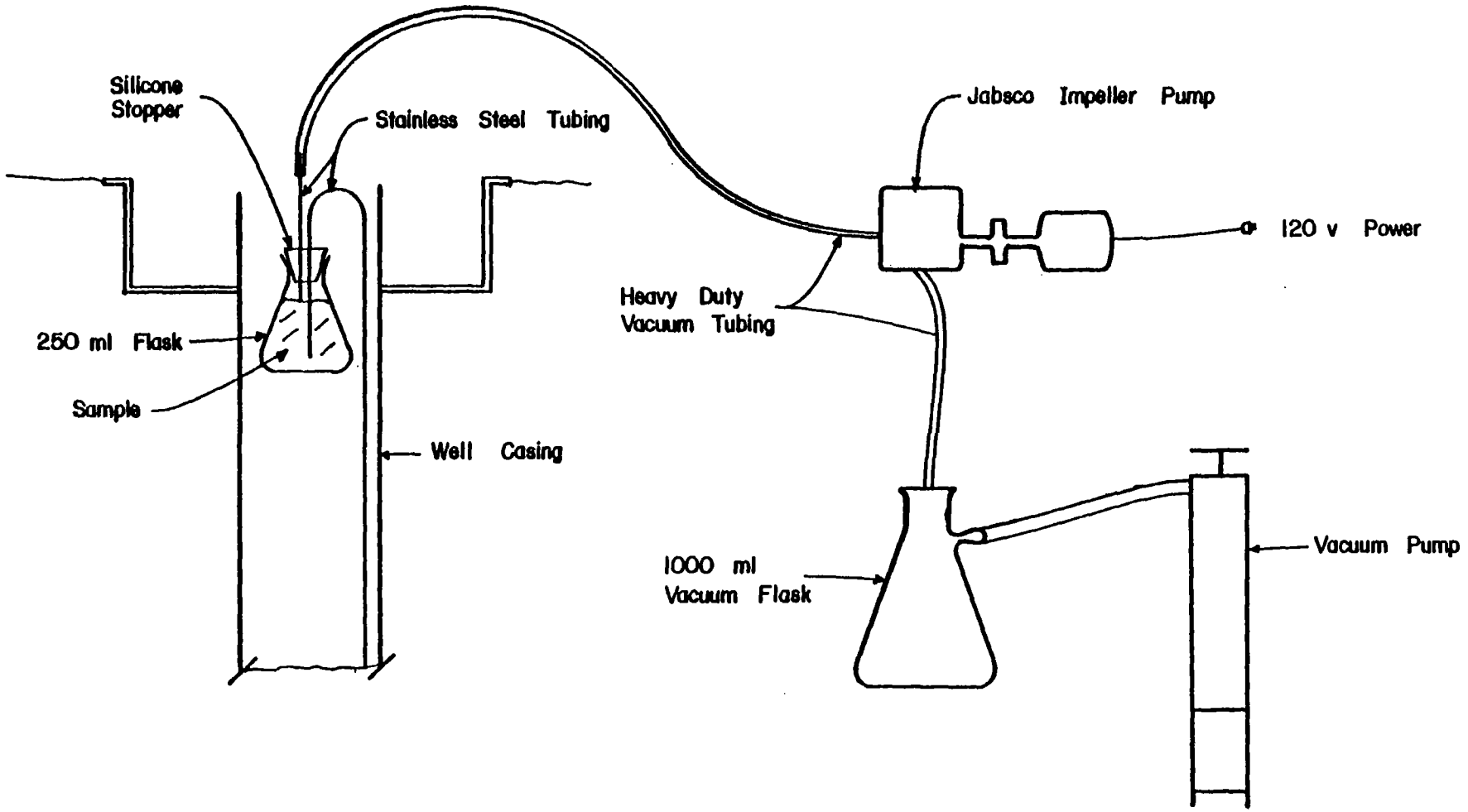


FIGURE 3

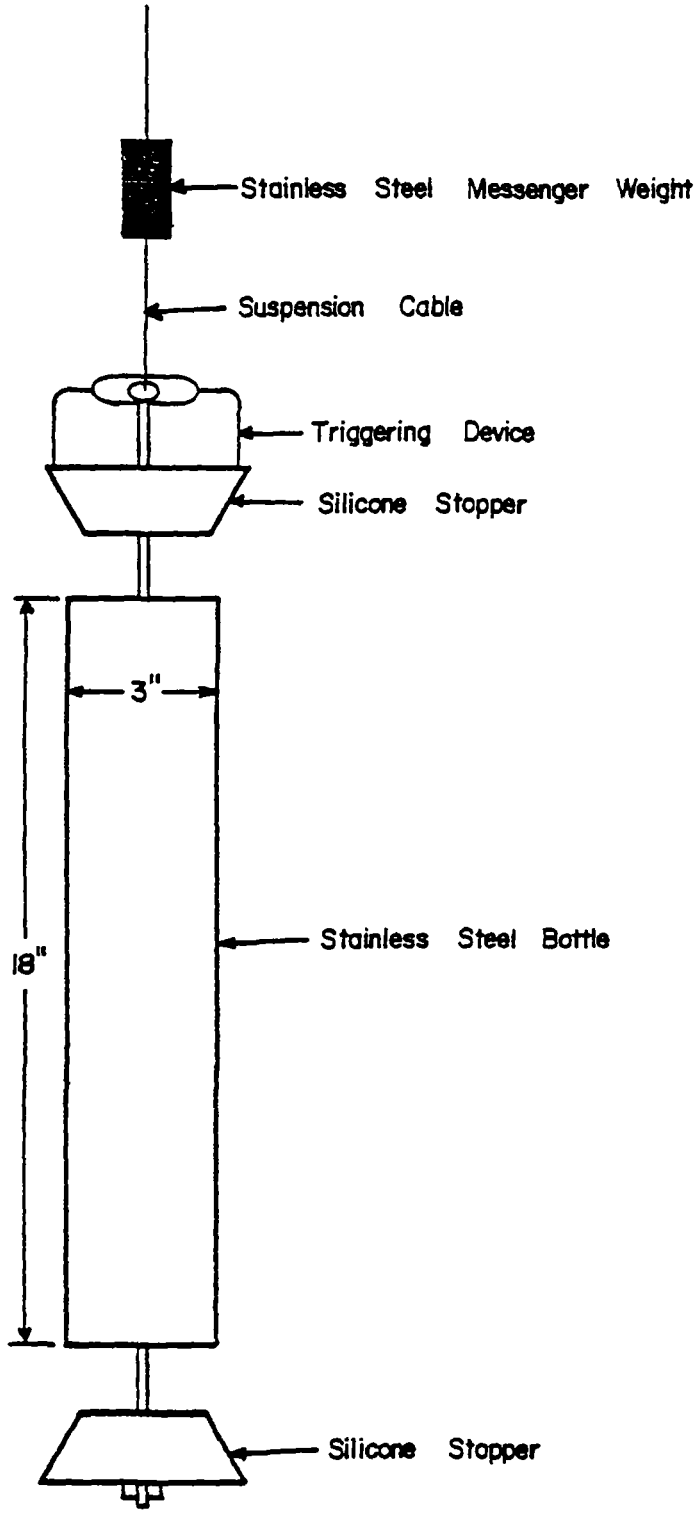
Depending on elevation, the flow rate varies from 0.75 gal./min. to 2.0 gal./min. These rates have been checked in the field. On most wells, flow rates average 1-1.5 gal./min. providing for removal of 10-15 gallons. (e.g., a well 25' deep with an elevation of 13.5' would have a flow rate of approximately 1.5 gal./min., and after 10 minutes of pumping would remove approximately twice the volume of the water in the well, see calculations p. 48).

After 10 minutes of pumping, the silicone stopper is removed from the flask while the pump is still running to avoid any runback from the pump. The sample vials are then filled and sealed without headspace from the flask. The flask is then emptied and thoroughly rinsed with methanol, inverted and allowed to dry. Four flasks are rotated from well to well to insure no cross contamination occurs. The flasks are thoroughly cleaned between sampling dates in the lab.

The cleaning of the flasks combined with flushing the sample flask for 10 minutes with 10-15 gallons of sample provides a "clean" representative sample from each well. It should be noted here that any air introduced to the sample while pumping (i.e., air bubbles which appear in the flask while pumping) cannot be eliminated by any kind of surface pumping system. The same amount of suction is required to lift the sample whether the pump is impeller, gear, peristaltic, or vacuum, etc.

On the wells with a water elevation greater than approximately 24 feet, head and tubing size restrictions make surface pumping impractical. On these wells, we use a specially made

KEMMERER BOTTLE SAMPLER



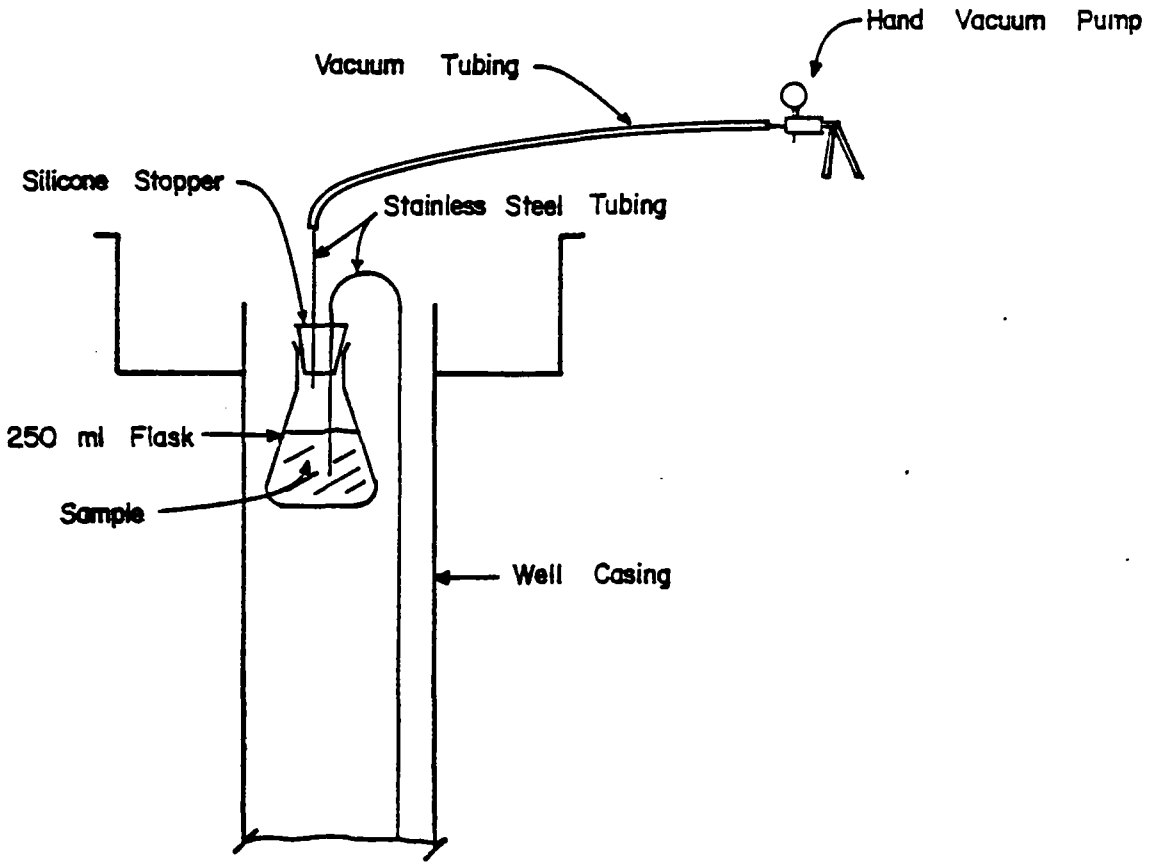
Kemmerer Bottle sampler, consisting of a stainless steel tube, with silicone stoppers at each end and steel cable (see Figure 4).

The sampler is lowered into the well and allowed to touch the bottom before being raised approximately 1 foot. A stainless steel messenger is dropped to trigger the sampler. The sampler is then withdrawn with a manually operated reel. Once removed from the well a sampling port at the bottom of the sampler is utilized to fill the vials in the same fashion as before. Care is taken to allow the sampling port to run briefly to flush it before filling the sample containers. Directly after use, the entire sampler and messenger weight are washed thoroughly with methanol, and placed on aluminum foil to dry.

On wells which are grossly contaminated (that is, visibly contaminated), sampling by pumping is not acceptable due to destruction of the pump by the sample and a problem with disposal with the pump discharge. For grossly contaminated wells with water elevations of less than 24 feet, therefore, a hand vacuum sampling system can be used. Kemmerer bottle or bailer apparatus may be utilized when sampling grossly contaminated wells, however EXTREME CARE must be exercised to thoroughly clean all surfaces which come in contact with the sample. Improper cleaning greatly increases the probability of users contamination.

Stainless steel tubing is installed in these wells in the same manner as described before. A flask and silicone stopper is connected to the tubing and the flask is evacuated with a hand vacuum pump (see Figure 5). The sample vials are filled from the flask, which is then rinsed several times with methanol and allowed to dry.

GROSSLY CONTAMINATED WELL
VACUUM SAMPLING



The samples are kept in an insulated ice cooler until they are turned into the lab for analysis. A record is kept of all water elevations and is recorded for computer input.

Example of Well Volume Calculation

7.48 gal./ft^3

$$\text{AREA OF 4" WELL} = \frac{\pi d^2}{4} = \frac{\pi(4")^2}{4} = 12.56 \text{ inc.}^2 \text{ OR } \underline{0.087 \text{ ft.}^2}$$

$$\text{LENGTH OF CASING TO} = 1 \text{ ft}^3 = 0.087 \text{ ft}^2) \times (\text{N ft.}) = 1 \text{ ft}^3$$

$$\underline{\text{N} = 11.5 \text{ ft.}}$$

∴ a well with a total depth of 25 ft. with a water elevation of 13.5 ft. would contain ≅7.5 gallons and at a flow rate of 1.5 gal./min. twice the volume of the water in the well would be removed.

CHAPTER 8

TAGGING AND CHAIN OF CUSTODY

A sample is physical evidence collected from a facility or from the environment. An essential part of all enforcement investigations is that evidence gathered be controlled. To accomplish this, the following sample identification (tagging) and chain-of-custody procedures are used.

SAMPLE IDENTIFICATION (TAGGING)

Samples other than in-situ measurements, are identified by a sample tag, or other appropriate identification

These samples are transported from the sample location to a laboratory or other location for analysis. Before removal, however, a sample is often separated into portions, depending upon the analyses to be performed. Each portion is preserved in accordance with the applicable procedures and the sample container is identified by a sample tag. Sample tags are completed for each sample, using waterproof ink. The information recorded on the sample tag includes:

Project Code	-	A number assigned by O'Brien and Gere
Station Number	-	A number assigned by the Project Coordinator and listed in the project plan or the NPDES permit number if used for NPDES inspections.
Date	-	A six-digit number indicating the year, month and day of collection.
Time	-	A four-digit number indicating the military time of collection - for example 0954.
Station Location	-	The sampling station description as specified in the project plan.
Samplers	-	Each sampler is identified.
Tag Number	-	A unique number is stamped on each tag

Remarks - The samplers record pertinent observations.

The tag used for water samples (also soil, sediment and biotic samples) contains an appropriate place for designating the sample as a grab or a composite, and identifying the type of sample collected for analyses and preservative, if any. The Project Coordinator will detail procedures for completing tags used for soil, water, sediment, and biotic samples. The sample tags are attached to or folded around each sample.

After collection, separation, identification, and preservation, the sample is maintained under chain-of-custody procedures discussed below. If the composite or grab sample is to be split, it is aliquoted into similar sample containers. Identical sample tags are completed and attached to each split and marked "Split." In a similar fashion, all tags on blank or duplicate samples will be marked "Blank" or "Duplicate" respectively.

CHAIN-OF-CUSTODY PROCEDURES

Due to the evidentiary nature of samples collected during enforcement investigations, possession must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. To maintain and document sample possession, chain-of-custody procedures are followed.

Sample Custody - A sample is under custody if:

1. It is in your possession, or
2. It is in your view, after being in your possession, or
3. It was in your possession and then you locked it up to prevent tampering, or
4. It is in a designated secure area.

Field Custody Procedures

1. In collecting samples for evidence, collect only that number which provides a good representation of the media being sampled. To the extent possible, the quantity and types of samples and sample locations are determined prior to the actual field work. As few people as possible should handle samples.
2. The field sampler is personally responsible for the care and custody of the samples collected until they are transferred or dispatched properly.
3. The Project Coordinator determines whether proper custody procedures were followed during the field work and decides if additional samples are required.

Transfer of Custody and Shipment

1. Samples are accompanied by a Chain-of-Custody Record, Figure 6. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another

Figure 6

O'BRIEN & GERE

CHAIN OF CUSTODY RECORD

SURVEY				SAMPLERS: *(Signature)*					
STATION NUMBER	STATION LOCATION	DATE	TIME	SAMPLE TYPE			SEQ. NO.	NO. OF CONTAINERS	ANALYSIS REQUIRED
				Water		Air			
				Comp.	Grab.				

Relinquished by: *(Signature)*	Received by: *(Signature)*	Date/Time
Relinquished by: *(Signature)*	Received by: *(Signature)*	Date/Time
Relinquished by: *(Signature)*	Received by: *(Signature)*	Date/Time
Relinquished by: *(Signature)*	Received by Mobile Laboratory for field analysis: *(Signature)*	Date/Time
Dispatched by: *(Signature)*	Date/Time	Received for Laboratory by:	Date/Time
Method of Shipment:			

O'Brien & Gere Engineers, Inc.
Box 4873 / 1304 Buckley Road / Syracuse, NY 13221 / (315) 451-4700 / CABLE OBRIENGERE
Boston, MA / New York, NY / Philadelphia, PA / St. Louis, MO / Washington, DC / White Plains, NY

person, to the analyst in a mobile laboratory, or at the laboratory.

2. Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment (for example, one for each field laboratory, one for samples driven to the laboratory). Shipping containers will be padlocked or sealed for shipment to the laboratory. The method of shipment, courier name(s) and other pertinent information are entered in the bottom of form.

3. Whenever samples are split with a source or government agency, it is noted in the "Remarks" section. The note indicates with whom the samples are being split and is signed by both the sampler and recipient. If either party refuses a split sample, this will be noted and signed by both parties. The person relinquishing the samples to the facility or agency should request the signature of a representative of the appropriate party, acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Remarks" space. When appropriate, as in the case where the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time.

4. All shipments will be accompanied by the Chain-of-Custody Record identifying its contents. The original record will accompany the shipment, and a copy will be retained by the Project Coordinator.

5. If sent by mail, the package will be registered with return receipt requested. If sent by common carrier, a Government Bill of Lading will be used. Air freight shipments are sent collect. Freight bills, Post Office receipts, and Bills of Lading will be retained as part of the permanent documentation.

FIELD FORMS

Appropriate field sheets must be completed at the time of sample collection. In addition, a bound field notebook must be maintained by the survey leader to provide a daily record of significant events. All entries must be signed and dated. All members of the survey party must use this notebook. Keep the notebook as a permanent record. In a legal proceeding, notes, if referred to, are subject to cross-examination and admissible as evidence.

GENERAL REFERENCES

1. Federal Register, Vol. 44, No. 233, December 3, 1979

2. Federal Register, Vol. 45, No. 98, May 19, 1980

3. U.S. EPA "Procedures Manual for Ground Water Monitoring at Solid Waste Disposal Facilities", August, 1977 (EPA/530/SW-611)

4. U.S. EPA "Methods for Chemical Analysis of Water and Wastes", March, 1979 (EPA - 600/4-79-020)

5. U.S. EPA "Handbook for Sampling and Samples Preservation of Water and Waste Water", September, 1982 (EPA-600/4-82-029)

6. 1981 Annual Book of ASTM Standards, Part 31, Method D-3370 "Standard Practices for Sampling Water".

7. "Standard Methods for the Examination of Water and Wastewater", 15th Ed. 1980 (APHA, AWWA, WPCF)

APPENDIX

VOLATILE ORGANIC COMPOUNDS (VHO, BTX, AND THM)

Sample Container

A special vial is used to sample Volatile Organic Compounds in water. The 40ml vial is made of high purity borosilicate glass and has a black plastic cap with a teflon lined silicon rubber septum. When properly filled and capped, the water sample contains no air bubbles (headspace) and contacts only teflon and glass.

General Procedure

1) Gently fill the 40ml vial without causing undue turbulance. Allow sample to overflow the vial filling it to its maximum capacity.
2) With the teflon septum inserted into the cap, place the cap on the vial and tighten. The teflon side of the septum is the side which is not soft rubber and the side which should contact the water sample.
3) Invert the bottle and tap on a hard surface to ensure no air bubbles are present. If they are, repeat the procedure.
4) Keep vials cool during shipment to laboratory.
5) Return to laboratory within 24 hours.
6) Record all pertinent information:
    a. Sample Site and Date
    b. Sample Identification
    c. Chain of Custody forms (if required)

EXTRACTABLE ORGANIC COMPOUNDS (PESTICIDES, PCBS, BASE/NEUTRALS/ACIDS, ETC.)

Sample Container

The sample container is a liter size glass bottle with either a teflon lined or aluminum foil lined cap. When properly filled and capped, the sample contacts only teflon and glass.

It is important during collection that the sample does not contact plastic. Contact with plastic may lead to erroneous laboratory results.

General Procedure

1) Fill the contents of sample bottle to the brim and cap. Do not rinse sample bottle with sample prior to filling.
2) Keep sample bottles cool during shipment to laboratory.
3) Return to laboratory within 24 hours.
4) Record all pertinent information:
    a. Sample Site, Date and Time
    b. Sample Identification
    c. Chain of Custody forms (if required)

SAMPLING WATER TAPS (FAUCETS)

When sampling from a tap, turn on water and allow the system to flush. When the temperature of the water has stabilized, reduce water flow and collect sample from the gently flowing stream.

General Procedure

1) Use a clean glass jar/bottle.
2) Turn on water and allow the system to flush for 3 to 5 minutes.
3) When the temperature of the water has stabilized, reduce the water flow.
4) If the sample is to be analyzed for VOLATILE ORGANIC COMPOUNDS AND/OR EXTRACTABLE ORGANIC COMPOUNDS refer to these sections (pages 57 and 58).
5) Tilt the sample bottle and collect sample from the gently flowing stream.
6) Properly preserve the sample (see Table I Chapter 2).
7) Store sample in an insulated ice cooler at 4°C.
8) Return sample to the O'Brien & Gere laboratory for analysis.
9) Record all pertinent information
    a. Sample site and date
    b. Sample identification
    c. Chain of Custody forms (if required)

SURFACE WATERS

When sampling from an open body of water, care must be exercised to collect a representative sample. In many cases, several preserved containers are required based on the analytical scheme. Therefore, a clean glass container is required to transfer the sample to the preserved containers. Collection is accomplished by inverting the container, submerge, and while submerged, upright the container allowing the air to escape. Slowly retrieve the container and transfer its contents to the preserved containers.

NPDES COMPLIANCE

Sampling for permit requirements is normally dictated on each permit. These permits apply to sanitary and storm sewers, treatment plant discharges and other point sources of pollution. Normally compliance samples are composited over the time of discharge which varies with each sewer. However grab samples are sometimes requested. The permit requirements should be received prior to sample collection.

The grab sample consists of an individual sample (approximately 100 to 200ml) collected at randomly selected times over a period of time not exceeding 15 minutes. The composite sample is comprised of a minimum of 8 aliquot samples (approximately 100ml each) collected at periodic intervals over a 24-hour period. The aliquots may be collected either automatically or manually. However, since the composite is proportional to flow, proper care must be used between aliquot collections such that the volume of each aliquot (or the time interval between each aliquot) must be proportional to either the stream flow at the time of sampling, or the total stream flow. The installation of proper flow monitoring equipment is paramount for compositing discrete grab samples.

General Procedure

1. Inspect manhole to verify:
    a. sufficient room for equipment,
    b. no restrictions in channel,

c. no other laterals. There should be only one inlet and one outlet.

2. Install flow monitoring equipment:
   a. Weir.
   b. Water level recorder.
3. Verify equipment is recording information by allowing to run unattended for 2 hours beginning to sample.
4. If conventional pollutants are to be analyzed (nutrients, heavy metals, demand substances) an automatic sampler may be utilized for collection of discrete samples. However if purgeable organics, cyanide, phenol, chlorine residual, or bacteria are required they must be collected manually due to immediate preservation.
5. To manually collect a sample merely place a clean glass quart jar into the flowing stream (mouth of jar terminal flow) and fill.
6. Return to surface, dispense sample into proper container and remaining sample will be used for compositing.
7. Continue collecting discrete grab sample every hour for 24 hours.
8. Place all samples in ice cooler maintained at 4°C.
9. Return all samples to lab for compositing. Samples preserved in the field for cyanide, phenol, chlorine residual or bacteria must be analyzed individually then averaged for composite value.

10. Discrete samples are flow proportioned to generate a composite sample. Purgeable organics are composited in a different manner.

    a. Place a 1000 ml round bottom flask in an ice bath.

    b. Carefully pour the entire contents of one vial down the side of the flash to minimize turbulance.

    c. After all discrete grabs have been added to flask, gently stir the mixture for one minute with a glass rod.

    d. Transfer a portion of the material to 4 purgeable vials for analyses. Follow, same collection procedures for volatile organic compounds.

11. Record all pertinent information.

    a. Sample site and date

    b. Sample identification

    c. Chain of Custody (if required)

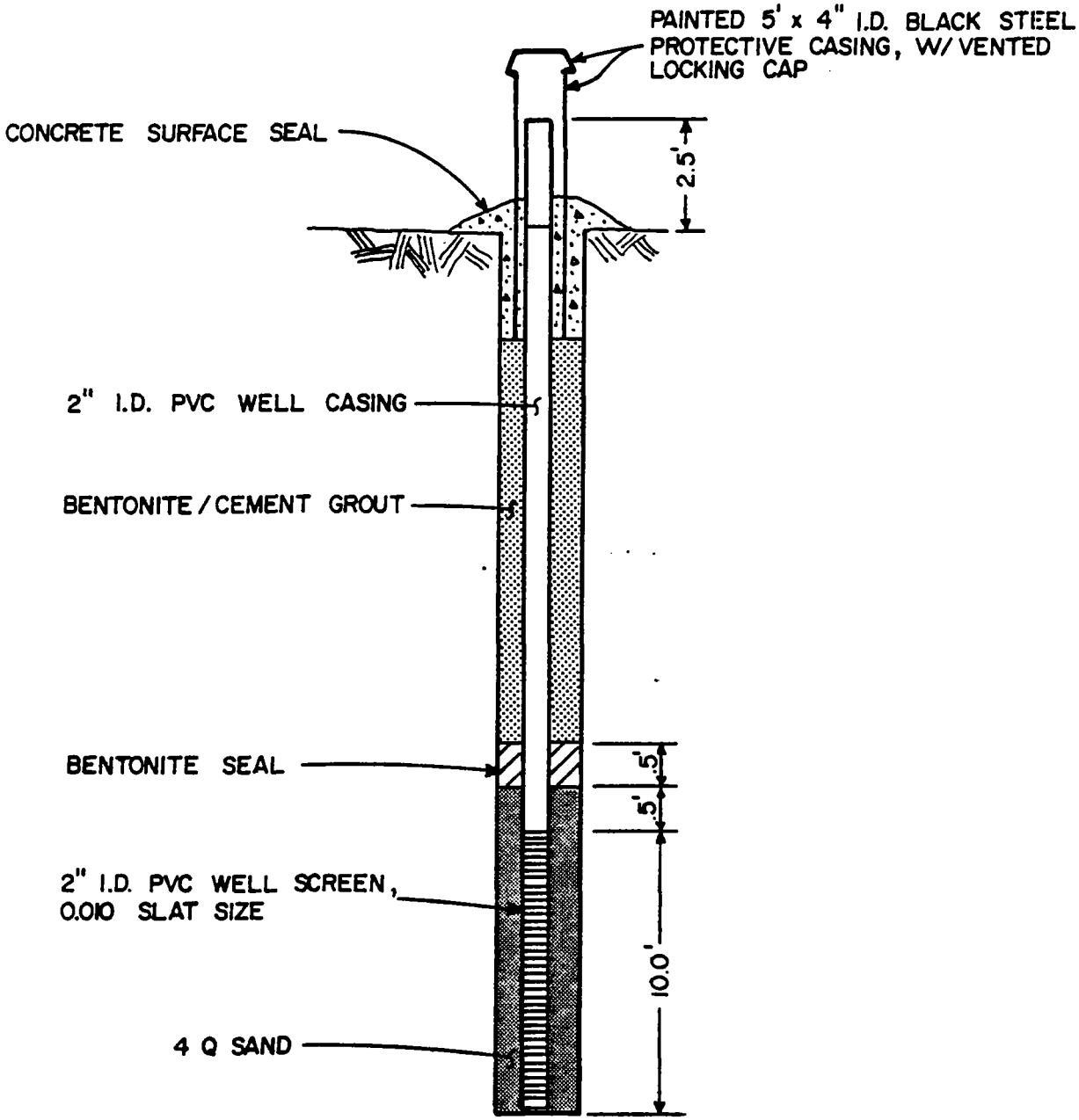
GROUNDWATER WELLS

When sampling a groundwater well care must be exercised to collect a sample which is uncontaminated and representative. Prior to collection, the well should be evacuated with 3 - 5 well volumes of water. After evacuation, the well should be allowed to recharge before collection. To accomplish collection, insert a stainless steel bailer Kemmerer apparatus, or vacuum system. Dispense the sample to proper containers. When collecting samples from several wells, be sure to use a new or clean sampler to minimize cross contamination.

General Procedure

1. Well Evacuation - with a pump.
    a.) Connect the top end of the stainless steel tubing (see Figure 2) to a gallon glass bottle with teflon tubing.
    b.) A second piece of stainless steel tubing is connected to a 120V "Jabsco impeller pump" with a piece of heavy-duty vacuum tubing.
    c.) A third piece of heavy-duty vacuum tubing is connected to the pump discharge and runs to a large 1000 ml vacuum flask. The flask is evacuated by a large capacity manually operated vacuum pump (only needed to assist pump when used on deeper wells).
    d.) The vacuum applied to the system with the vacuum pump draws water up the tubing into the first (250 ml) flask and through the impeller pump which in effect primes the pump.

TYPICAL MONITORING WELL



e.) The primed impeller pump is started and the large (1000 ml) vacuum flask is removed.

f.) The pump is allowed to run for ten minutes (or until 3 to 5 well volumes of water are evacuated).

g.) Collect the discharge water in a 5 - 10 gallon receptical and transport to treatment facility for disposal.

h.) After ten minutes evacuation the well is allowed to recharge before sample collection.

2. Well Evacuation - Bailer

a.) Lower clean bailer or Kemmer assembly (Figure 3) into well until the entire apparatus is submerged.

b.) Trip assembly for sample collection and redraw the equipment.

c.) Dispense the contents into receptacle for disposal.

d.) Repeat steps a) through c) until 3 - 5 well volumes have been removed.

e.) Allow well to recharge before collecting sample.

3. Wells With Water Elevation Less Than 24 Feet

To obtain a water sample from the groundwater well first follow procedure 1 above. Then, after 10 minutes of pumping:

a.) Remove the silicone stopper from the 250 ml flask while the pump is still running.

b.) Use the water sample in the 250 ml flask to fill four clean 40 ml glass vials which are then sealed without headspace.

WELL PUMPING - SAMPLING SYSTEM

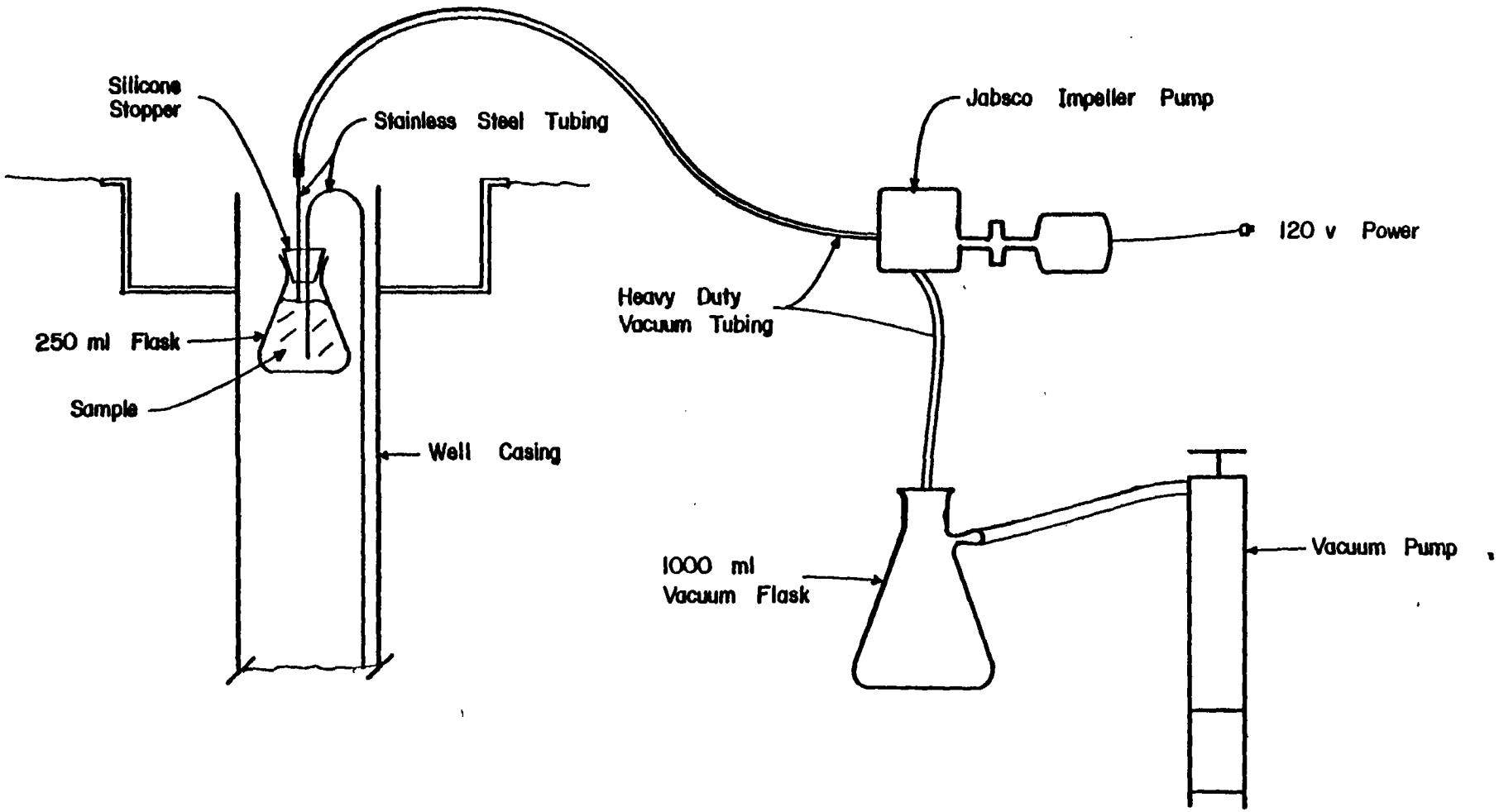


FIGURE 2

c.) Store sample in an insulated ice cooler at 4°C.

d.) Return samples to the O'Brien & Gere laboratory for analysis.

e.) Record all pertinent information.

1. Sample Site and Date
2. Sample Identification
3. Chain of Custody (if required)

4. Wells With Water Elevation Greater Than 24 Feet

On wells with water elevation greater than ca. 24 feet, head and tubing size restrictions make surface pumping impractical. One these wells, a Kemmerer Bottle sampler or bailer is used (see Figure 3). To obtain the water sample:

a.) Carefully lower the sampler into the well until it touches bottom.

b.) Then raise the sampler up approximately 1 foot.

c.) Drop the stainless steel messenger weight to trigger the sampler.

d.) Withdraw the sampler with a manually operated reel.

e.) The sampling port at the bottom of the sampler is used to fill the glass vials. (Care is taken to allow the sampling port to run briefly to flush it before filling the sample vials).

f.) Store samples in an insulated ice cooler at 4°C.

g.) Return samples to the O'Brien & Gere laboratory for analysis.

h.) Record all pertinent information

1. Sample Site and Date
2. Sample Identification
3. Chain of Custody (if required)

i.) Directly after use, thoroughly wash with methanol the entire sampler and messenger weight, and place it on aluminum foil to dry.

5. Grossly Contaminated Wells

When sampling grossly contaminated wells, a hand vacuum sampling system is used. To obtain the water sample:

a.) Connect a clean 250 ml flask and silicone stopper to the stainless steel tubing (see Figure 4).

b.) Insert a second piece of stainless steel tubing through the silicone stopper and connect it to a hand vacuum pump with a piece of vacuum tubing (see Figure 4).

c.) Evacuate the flask with the hand pump and collect the sample.

d.) Fill the glass vials with the sample from the flask.

e.) Store the samples in an insulated ice cooler at 4°C.

f.) Return samples to the O'Brien & Gere laboratory for analysis.

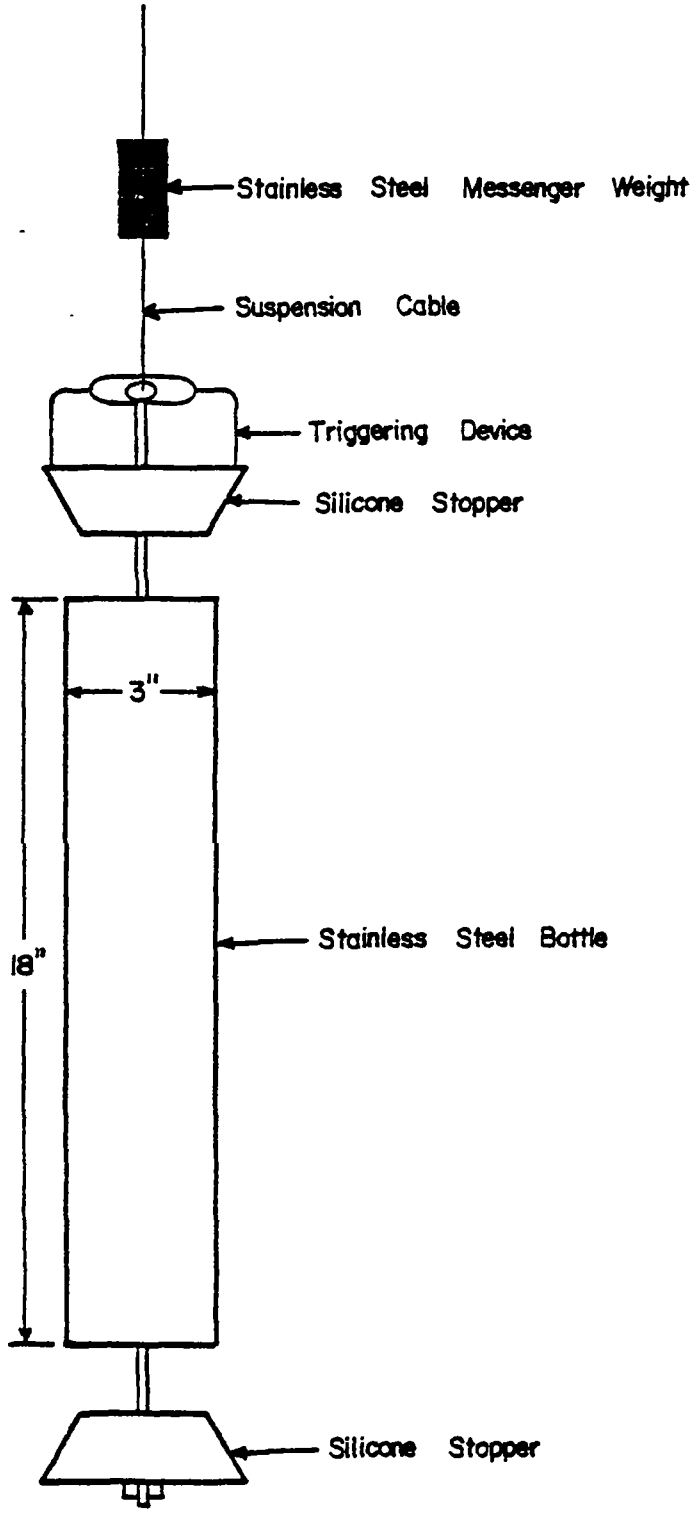
g.) Record all pertinent information

1.) Sample Site and Date

2.) Sample Identification

3.) Chain of Custody (if required)

KEMMERER BOTTLE SAMPLER



ADDENDUM

SAMPLING SOILS

BACKGROUND

Surface runoff groundwater recharge normally transport and materials from soils to surface waters or groundwater. These soil surfaces are often exposed to contaminated fluids and solids. Consequently it is often necessary to determine whether the soils near the surface have a reservoir of contaminants which will migrate throughout the environment through such mechanisms as groundwater recharge and surface runoff.

VOLUME OF SAMPLE AND CONTAINER TYPE

Refer to Chapter 2 for specific information relative to the chemical parameters which are to be analyzed. If possible do not use metal samplers for trace metals or plastic samplers for trace organics.

PRESERVATION AND HANDLING OF SAMPLES

Refer to Chapter 2 for specific information regarding preservation and handling of samples. Soil samples can not be readily preserved due to the difficulties associated with exposing the entire samples to an appropriate preservative. Where conventional non volatile components are to be determined the sample should be placed in an appropriately sized wide mouth glass container with a teflon cap liner. After the cap

has been placed on the container it should be sealed with wax to insure minimal change in soil composition during transport for analysis. When volatile organics such as Trichloroethylene are to be analyzed, the laboratory supervisor should be contacted well in advance of the sampling program to obtain the specialized containers required for this application.

SAMPLE COLLECTION

Surface Soil Samples

A polypropylene or aluminum scoop can be used to collect surface soil samples. Where trace organic contamination is a concern then the aluminum scoop should be used.

An appropriate sample area usually less than one square foot per sampling location should be identified. At regular intervals small equal portions of the surface soil should be sampled. The small samples should be combined in a suitable container. The container should be capped, labeled, sealed and appropriate paper work completed prior to moving to the next sample site. It should be noted that the scoop should be decontaminated between sites. Appropriate solvents will be determined by the project objectives. Typically, a detergent wash followed by a rinse with acetone and air drying is adequate.

Soil Samples Within Four Feet of Grade Elevation

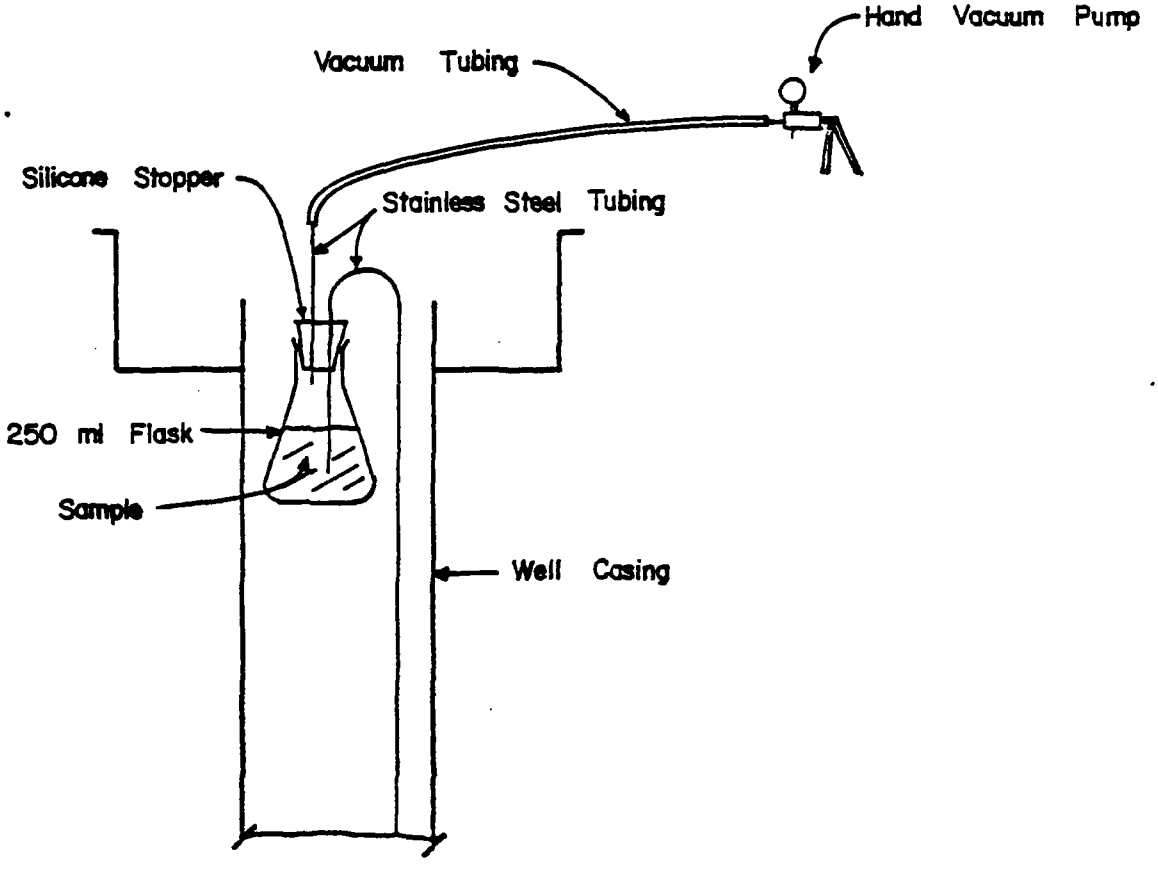
The sampling equipment required will depend to a great extent on the consistency of the soil being sampled. Among the tools which can be used are: soil auger, split barrel sampler, Wildco hand operated core sampler and Lexan tubing.

Where disturbed samples are adequate, the auger is a suitable tool. After selecting a sample location, the area should be cleared of unnecessary rocks twigs and other non-soil materials. The auger should be assembled and a hole bored through an aluminum pie pan large enough to allow the blades of the auger to pass through. The pan should be located against the selected sampling point. Start augering through the hole in the pan until the desired sampling depth is reached. Back off the auger and transfer the sample collected in the catch pan and the sample adhering to the auger to a suitable container. Spoon out the rest of the loosened sample with a sampling trier. If a larger volume of sample is required, repeat as required. Seal the container as described for the scoop sampler.

A split barrel sampler is available for use where core samples of approximately 18 inches are required. The sampler is provided with a driving rod and can be operated at depths as great as 48 inches. If samples are required at a depth greater than 18 inches below grade either a shovel or power auger with a bit size of eight inch diameter should be used to provide an open hole to the elevation to be tested. The auger should be stopped at a minimum of 6 inches above the sampling level. After driving the split spoon to the required depth the spoon puller should be used to extract the split spoon. The split spoon should be opened and the undisturbed sample classified and

FIGURE 4

GROSSLY CONTAMINATED WELL
VACUUM SAMPLING



placed in appropriate containers. The split spoon will require decontamination between sampling sites as described for the scoop sampler.

Two Wildco hand operated core samplers are available for use in soil sampling. One unit will provide an undisturbed sample with a maximum depth of 3½ feet while the other is designed for 18 inch samples. After assembling the unit, the sampler is manually advanced into the soil or sediments until the desired depth is reached. The sampler is rotated 360 degrees and removed. It is often necessary to use a tripod with a winch to remove the assembled sampler. Once removed, the nose piece for the core sampler is removed and the 2 inch outside diameter Lexan® liner is removed. If sampling is in a moist location care should be taken to maintain the sampler in an upright position. Tubing caps should be placed on the Lexan® and sealed with wax. The tubes should be labeled and shipped to the laboratory in an upright position for sectioning and analysis. The assembly must be decontaminated between samples.

In moist locations with unconsolidated sediments, the use of Lexan® tubing without an exterior casing is an effective sampling method. Tubing with an interior diameter ranging from 1-1/4 to 1-7/8 inches are often used. The tubing may be driven into the sediment using hand tools to the desired depth. In soft sediments, samples of up to eight (8) feet may be obtained. However, due to consolidation, this method generally results in obtaining samples of shorter length. Records of water elevation, sediment elevation, bottom of coring elevation and core length should be maintained. Samples in Lexan® tubing should be handled in the same manner as described above.

Work Plan Appendix C
Site Health and Safety Plan

Remedial Investigation/ Feasibility Study

Crab Orchard National Wildlife Refuge

U.S. Fish and Wildlife Service
U.S. Department of Interior
Marion, Illinois
and
Sangamo-Weston, Inc.
Atlanta, Georgia

June 1985

APPENDIX C

SITE HEALTH AND SAFETY PLAN

REMEDIAL INVESTIGATION/FEASIBILITY STUDY

CRAB ORCHARD NATIONAL WILDLIFE REFUGE

U.S. FISH AND WILDLIFE SERVICE

U.S. DEPARTMENT OF INTERIOR

MARION, ILLINOIS

AND

SANGAMO-WESTON, INC.

ATLANTA, GEORGIA

O'BRIEN & GERE ENGINEERS, INC.
1304 BUCKLEY ROAD
SYRACUSE, NEW YORK 13221

JUNE 1985

TABLE OF CONTENTS

SECTION 1 - INTRODUCTION AND BACKGROUND INFORMATION

This document is the Site Health and Safety Plan for site activities to be conducted during the Remedial Investigation/Feasibility Study (RI/FS) being performed at the Crab Orchard National Wildlife Refuge by O'Brien & Gere Engineers.

All personnel (here defined as employees of O'Brien & Gere Engineers, employees of all subcontractors, respondents, all visitors and representatives from the EPA, State, local groups, media, etc.) will be required to follow and adhere to the procedures set forth in this plan. All personnel will also be required to report to the Site Health and Safety Officer (SHSO) before proceeding on-site.

The RI/FS of the Site will involve many complicated operations conducted over 30 months duration. The need for Task Specific Health and Safety Plans to be developed will be determined on a Site walk-through scheduled for July 15, 1985. If such are deemed appropriate, then they will be included as Attachments hereto prior to the initiation of major Tasks.

1.01 Identification

Site Name: Crab Orchard National Wildlife Refuge

Address/Location: Marion, Illinois 62959

Project Description: Remedial Investigation/Feasibility Study (RI/FS)

On-Site Work Dates: July 1985 through June 1986

Overall Degree of Hazard: Low to Moderate

1.02 Key Personnel for RI/FS

U.S. EPA Contact:	Rodney Gaither (312) 886-4735
U.S. Fish & Wildlife Service Contact:	Dick Ruelle (309) 793-5800
Refuge Contacts:	Wayne Adams (618) 997-3344
O'Brien & Gere Contact:	Cornelius B. Murphy (315) 451-4700
Task & Safety Coordinator:	Swiatoslav Kaczmar (315) 451-4700

1.03 Site Description

Type of Facility: Wildlife refuge and waterways.

Size: Study area includes approximately 30,000 acres.

Buildings: Various (office, storage, industrial).

Surrounding Land Uses: Residential and rural.

Layout: The site is located on Crab Orchard Lake on the southwest border of Marion, IL.

1.04 Site History

The primary use of many of the refuge sites was the manufacturing, loading and storage of explosives. In addition, the manufacture of nitrogen fertilizer also took place. There are indications that waste disposal by landfilling was practiced at a number of the facilities. Current use and/or status of the site includes explosives manufacturing, chemical manufacturing and pen/ink/stencil production. Effluent discharge into refuge waterways from local facilities is suspected. The remainder of the sites reviewed stand as landfills, waste disposal sites and impoundment dikes.

1.05 Summary of Site Hazards

The site hazards include (but as yet determined are not limited to):

- fire and/or explosion hazard due to presence of explosives (possibly including lead azide and RDX-cyclonite);
- direct contact and environmental hazards from the possibility of leaks from the nitric acid pond;
- environmental and/or human risk due to unidentified barrels of chemical waste;
- explosion hazard regarding the impoundment dikes; and
- persistence of PCP (pentachlorophenol) remains from prior use as a wood (telephone pole) preservative.

Table 1, "Summary of Site Hazards", summarizes the anticipated hazards associated with remedial investigation work at each individual site. Table 1 was compiled based on information collected during the initial site walk-through. An additional safety and health survey will be conducted prior to initiation of field activities. That survey will be consistent with the forms included in Appendices B and C of Attachment 1 (the Safety Manual).

1.06 Project Description and Purpose

The Remedial Investigation (RI) will determine the nature, extent and concentration of on-site wastes and environmental contaminants. It will also determine the degree of risk to human health and the environment which those contaminants represent. Most of the on-site work for the RI/FS will be conducted during the RI.

TABLE 1

SUMMARY OF SITE HAZARDS

GROUP	SITE #: SITE DESCRIPTION	POSSIBLE HAZARD LEVEL	SPECIFIC HAZARD CONCERNS
#1	3:AREA 11 SOUTH LANDFILL	HIGH	Abondoned explosives manufacturing site, Surrounding areas have a history of fires and explosions, powder canisters observed on site
	4:AREA 11 NORTH LANDFILL	HIGH	Possible storage site of explosives
	5:AREA 11 ACID POND	LOW	Acid pond impoundment of process wastes
#2	7A:D AREA NORTH LAWN	LOW	Stream, water and sediment sampling
	11A:P AREA NORTH	MODERATE	Possible surface deposits
	7:D AREA SOUTHEAST DRAINAGE	LOW	Stream, water and sediment sampling
	8:D AREA SOUTHWEST DRAINAGE	LOW	Stream, water and sediment sampling
	9:D AREA NORTHWEST DRAINAGE	LOW	Stream, water and sediment sampling
	10:WATERWORKS NORTH DRAINAGE	LOW	Stream, water and sediment sampling
	11:P AREA SOUTHEAST DRAINAGE	LOW	Stream, water and sediment sampling
	20:D AREA SOUTH	LOW	Stream, water and sediment sampling
#3	12:AREA 14 LANDFILL	LOW-MODERATE	Possible surface deposits of chemical wastes
	13:AREA 14 CHANGE HOUSE SITE	LOW-MODERATE	Former change house site may include spillage residues from munitions manufacturing
	14:AREA 14 SOLVENT STORAGE	LOW	Stream, water and sediment sampling
#4	15:AREA 7 PLATING POND	LOW	Pond water and sediment sampling
	16:AREA 7 INDUSTRIAL SITE	LOW	Sampling of surface soils
#5	17:JOB CORPS LANDFILL	LOW	Possible presence of PCB's. Other wastes observed appear to be mainly sanitary refuse
#6	18:AREA 13 LOADING PLATFORM	LOW-MODERATE	Sampling of surface soils. Possible presence of explosive chemicals
	19:AREA 13 BUNKER 1-3	LOW	Sampling of surface soils
	30:MUNITIONS CONTROL SITE	LOW	Explosives Control site - assumed to beclean
#7	21:SOUTHEAST CORNER FIELD	LOW	Surface soil sampling
#8	22:OLD REFUGE SHOP	LOW	Stream, water and sediment sampling
	24:PEPSI-WEST	LOW	Stream, water and sediment sampling
	25:C.O. CREEK AT MARION LF	LOW	Stream, water and sediment sampling
	26:C.O. CREEK BELOW MARION STP	LOW	Stream, water and sediment sampling
	27:C.O. CREEK BELOW 157 DREDGE	LOW	Stream, water and sediment sampling
#9	28:WATER TOWER LANDFILL	MODERATE-HIGH	Previous analysis for lead suggest that explosives residuals may be present
#10	29:FIRE STATION LANDFILL	HIGH	History of fires and known disposal of magnesium powder
#11	32:AREA 9 LANDFILL	HIGH	Previous analysis for lead suggest that explosives residuals may be present. PCB's known to be in soil
	33:AREA 9 BUILDING COMPLEX	MODERATE	PCB's known to be in soil
#12	34:CRAB ORCHARD LAKE	LOW	Biota, water and sediment sampling
#13	31:REFUGE CONTROL SITE	LOW	Refuge control - assumed to be clean

The Feasibility Study (FS) will identify and evaluate the appropriate remedial actions for the site, based on existing data and information gathered during the RI. This work phase is primarily engineering design evaluation and is to be conducted off-site.

The Tasks to be conducted on-site during the RI are fully described in the Work Plan.

SECTION 2 - HAZARD EVALUATION

2.01 Previous Monitoring Performed On-Site

A partial summary of published and unpublished monitoring data and information on Crab Orchard Creek Watershed and Crab Orchard Lake is included here as Attachment 2 in the Scope of Work for the Remedial Investigation/Feasibility Study.

During the initial walk-through, monitoring was performed using an HNU PI 101 photoionization detector and a Century OVA 128 organic vapor analyzer. No organic vapors were detected during any portion of the initial walk-through.

2.02 Previous Levels of Personnel Protection

Level D was used for the initial site review. Respiratory protection was available for immediate deployment, if necessary.

2.03 Hazardous Materials Known to be On-Site

Hazardous materials known to be on the NWR site include (but are not limited to):

- explosives and explosives residues
- nitric acid
- pentachlorophenol
- unidentified chemical wastes
- polychlorinated biphenyls (PCB's)
- magnesium dust
- lead dust

2.04 Overall Degree of Hazard

Low to Moderate

Air monitoring is recommended during boring or excavation. Immediate availability of air purifying respirators (organic vapor and particulate cartridge) must be maintained at all times for all personnel on-site during the above activities.

2.05 Specific Hazards

The presence of highly explosive materials necessitates extreme caution regarding flame, heat or other sources of ignition.

2.06 Respiratory Protection (RP) Action Levels

Level D - (no respiratory protection necessary) is expected to be used during most activities on the site. Monitoring of the site using a calibrated HNU-PI 101 photoionizing air monitor and/or Ova 128 organic vapor analyzer will be employed during field activities. Any organic vapor level reading showing an elevation of 2 ppm above background will be cause for an upgrading to Level C-RP.

Level C-RP - Air purifying respirator, high efficiency organic vapor/particulate filter cartridge will be available to all site personnel who have been fit-tested. Level C-RP will also be employed in dusty conditions, or during specific tasks which could result in a moderate, sudden release of vapor or dust. Any ambient

organic vapor level above 5 ppm will be cause for cessation of activities, and investigation into the implementation of a Level B respiratory protection plan.

Level B - Requires the use of a self-contained breathing apparatus (SCBA). Activities requiring this level of protection will not be pursued until a specific Level B Respiratory Safety Plan has been implemented.

2.07 Contact Protection

General dress requirements (minimum requirements for Level B, C, D) for work in designated contaminated zones are:

1. Rubber safety boots or safety work boots and rubber over boots (B, C & D).
2. Cotton coverall (D) or work clothing under white tyvek suit (B & C).
3. Tyvek hood (B & C).
4. Cotton gloves (D) or Surgeon's gloves and rubber over gloves (B & C).
5. Protective eyewear.
6. Hard hats during drilling.
7. Noise protection during drilling.

Specific tasks (sampling, test pits, and test borings) may require an upgrade to chemical resistant clothing - yellow tyveks, PVC coveralls, or butyl aprons. This will be determined by the SHSO or his designee during field activities.

SECTION 3 - PROTOCOLS FOR ROUTINE ACTIVITIES

3.01 Health and Safety Management and Responsibilities

Swiatoslav Kaczmar (O'Brien & Gere) -

Dr. Kaczmar will act as the Site Health and Safety Officer (SHSO). He will have responsibility for the safety of operations and the health and safety of all contractor personnel. He may designate an authorized representative to carry out and monitor compliance with these established safety protocols.

Subcontractors and Government Oversight Personnel -

All subcontractors are required to adhere to the requirements of this Site Health and Safety Plan. They may upgrade their level of personal protection where necessary in order to comply with their own corporate Health and Safety requirements.

During performance of large tasks, subcontractors may wish to designate one of their trained personnel as a Safety Officer. This person must coordinate actions with the Site HSO or On-Site Coordinator (OSC). It is recognized that all subcontracted Safety Officers are subordinate to the OSC and HSO as designated above.

3.02 General Requirements for Entry in Contaminated Zones

A contaminated zone is an area, with a defined perimeter constituting a potential environmental or human health hazard, on which work is to be conducted. One specific location on the perimeter will be designated as the Entry and Exit (E & E) Point for the contaminated

zone. Any passage of personnel and/or equipment onto or off of the contaminated zone is permitted only through the E & E Point.

Before proceeding onto the site past the Entry and Exit Points, all O'Brien & Gere and subcontractor personnel shall:

1. Be advised of the Health and Safety Plan, instructed in safety procedures and proper use of equipment and aware of potential hazards.
2. Be properly dressed and equipped.
3. Notify the OSC or SHSO.
4. Have medical approval for use of all safety equipment.

All personnel entering into areas or performing Tasks requiring Level C or B respiratory protection shall:

1. Have been fit tested and have medical approval.
2. Be clean shaven in areas where the mask touches the face.
3. Have had necessary respiratory training.
4. Work in a minimum of 2 person team.

3.03 Site Entry and Exit (E&E) Procedures

Entry procedures are as follows:

1. All personnel shall dress in required safety clothing and activate necessary monitoring equipment.
2. All personnel (or team/Task leader) notify the SHSO of intended operations.
3. OSC or HSO reviews team personnel with respect to Section 3.02 above.
4. Entry time and personnel are logged.

5. Team proceeds through the designated, controlled E&E point (entry and exit).

Exit procedures are:

1. All personnel exit through the designated E&E point.
2. All personnel go through appropriate decontamination corresponding to level of protection used. (See Appendices A and F of Attachment 1)
3. All personnel are logged out and time recorded.

3.04 Daily Start-up and Shut-down Procedures

Start-up procedures are:

1. SHSO reviews site conditions with respect to modifications of work and safety plans.
2. Personnel and team briefing, review and update of safety procedures.
3. Check out of safety and monitoring equipment.
4. SHSO ensures that the first aid station is operable.
5. SHSO initiates appropriate monitoring.
6. Site Entry Procedures (3.03) are followed.

Shut-down procedures are:

1. All personnel exit and decontaminate.
2. SHSO logs all personnel out.

3. When appropriate, the SHSO and OSC performs a site walk to ensure that all personnel are off site and that the site is secure.

4. Equipment and site are secured.

3.05 Action Levels/General Personnel Protection Guidelines

Dress requirements may vary from Task to Task. Minimum dress requirements are outlined in Section 2.07. Respiratory protection requirements are outlined in Section 2.06.

1. Level C (full-face respirator, high efficiency organic vapor/particulate/pesticide cartridge) Action Levels

Level C will be required during performance of Tasks in zones in which organic vapor concentrations exceed established thresholds. Level C requires continual air monitoring of work areas with a portable organic vapor analyzer.

2. Level D (no respiratory protection):

Level D will be allowed in contaminated zones not requiring respiratory protection as outlined above, and in support and clean areas. Level D requires an air monitoring program in down range and/or work areas.

3.06 Heat/Cold Stress

During weather above 70° or any situation of high humidity, workers will be routinely observed for symptoms of heat stress. Heat

stress will be prevented by periodic test breaks and the availability of cold fluids. At cold temperatures (below 40°F) workers will be required to wear adequate warm, dry clothing.

3.07 Decontamination Procedures

The following information is more fully described in Attachment 1 to this SHSP.

Personnel

As a minimum, all personnel exiting a contaminated zone will go through the following decontamination:

1. Boot wash (detergent or water)
2. Boot rinse (water)
3. Outer glove wash (detergent and water)
4. Outer glove rinse
5. Removal of boots, tyveks and then gloves.

All personnel shall be free of visible contamination prior to leaving the site.

Sample Containers

After obtaining the sample, all containers will be decontaminated with a detergent/water wash and water rinse. Waste samples may require an additional decontamination with acetone, methanol, or other non-priority pollutant solvents.

Sampling Equipment

All reusable sampling equipment (bailers, buckets, augers, split spoons, etc.) will undergo the following decontamination prior to initial use on site, between each use, and upon final use. Equipment shall be cleaned of all visible contamination.

1. Thorough detergent/water rinse.
2. Tap water rinse.
3. Solvent wash/rinse (methanol, or other nonpriority pollutant).
4. De-ionized/distilled water rinse.

After decontamination, sample equipment shall be placed in clean plastic bags or other suitable wrapping to prevent recontamination.

Geotechnical Apparatus

All geotechnical apparatus such as augers, rods, drill bits, casings, etc., and backhoe buckets (where used to excavate test pits for sampling) will undergo the following decontamination prior to use on-site, between each use on-site, and prior to removal from the site to remove all visible contamination and soils:

1. High pressure hot water (tap water) wash and/or steam cleaning (steam jenny).

Heavy Equipment

All trucks, drill rigs, backhoes, or other equipment will undergo decontamination prior to leaving the site. The decontamination, as a minimum will require a high pressure hot water (tap water) wash and/or a steam cleaning of tires and treads to remove all visible muck, soils and contamination.

SECTION 4 - EMERGENCY INFORMATION

4.01 Emergency Telephone Numbers

State Police: (618) 542-2171 (in DuQuoin)

Fire Department: (618) 993-2211

Ambulance Service: (618) 993-3019 or 997-4915

Hospital: 997-5341 (Marion Memorial Hospital)

Poison Control Center: 1-800-252-2020 (in Springfield)

° State your name, location and nature of emergency

° For Hospital Victim:

-Name and phone of family or emergency physician.

-Description of incident - chemicals involved, symptoms, nature of injury, proposed treatment, plan of transportation.

4.02 Directions to Hospital

From north of the lake:

Main Street into Marion (east)

Turn right onto Vicksburg St.

Hospital is there on the corner

From south/east of the lake:

Rte. 57 north to Main St.

East on Main to Vicksburg St.

Right onto Vicksburg

Hospital is on the corner

4.03 Procedures for Serious Injury/Exposure

1. Perform necessary emergency first aid.
2. Evacuate all personnel from area if dangerous.
3. Notify SHSO and OSC.
4. Call appropriate emergency support.
5. Perform secondary first aid and prepare victim for transport.
6. Evacuate victim to hospital.
7. Notify hospital of the incoming patient and type/severity of injury and exposure.

4.04 Procedures for Fire

1. Isolate the location of the fire and alert on-site personnel.
2. Evacuate all personnel from the area.
3. If possible, contain the fire. A fire extinguisher will be available at the entry and exit point(s).
4. Notify the fire department.

4.05 Contingency Plan

Signal - 5 one-second blasts of auto or air horn.

Action - All personnel immediately evacuate downrange areas and report to the site access point/decontamination line for instruction.

SECTION 5 - FIRST AID FOR EXPOSURE

The following is a general description of first aid measures to be employed on site. In all cases of symptoms of chemical exposure, first aid treatment is to be followed by full medical examination.

5.01 Inhalation

Symptoms: dizziness, nausea, lack of coordination, headache, irregular rapid breathing, coughing, choking, weakness, loss of consciousness, coma.

Treatment:

1. Bring victim to fresh air. Rinse eyes or throat if irritated.
2. Be prepared to administer CPR.
3. Evacuate victim to hospital.

5.02 Dermal

Symptoms: Same as above. Solvents may product irritation, rash, or burning.

Treatment:

1. Flush affected area with cool water for 5 minutes.
2. Cover with a clean dressing.
3. If central nervous system symptoms develop, evacuate victim to hospital.
4. Monitor victim for at least 48 hours.

5.03 Ingestion

Symptoms: Same as above, with stomach cramps.

Treatment:

1. Evacuate victim to hospital.
2. Do not induce vomiting.

5.04 Eye Contact

Symptoms: Redness, irritation, pain, impaired vision.

Treatment:

1. Flush with water for at least 5 minutes using a portable eyewash unit.
2. If severe, evacuate victim to a hospital.

ATTACHMENT 1

SAFETY MANUAL

SAFETY MANUAL
TABLE OF CONTENTS

NOTE: All Appendices are adapted from National Water Well Association "Groundwater Investigations at Hazardous Materials Sites": Safety Short Course

I. INTRODUCTION

Every employer is responsible for maintaining safe working conditions for his employees. The purpose of this manual is to provide O'Brien & Gere personnel with a basic framework for safe conduct in performing hazardous waste site investigations. The manual is designed to provide guidance to managing engineers, project managers, project engineers and technicians who will be involved in conducting site surveys, sampling activities and overseeing remediation activities.

This guidance package has been adapted from the United States Environmental Protection Agency "Safety Manual for Hazardous Waste Site Investigations" as published by the Office of Enforcement on September 19, 1979. The procedures have been adapted to meet the general needs of O'Brien & Gere and may be of some use to potential subcontractors for the range of hazardous waste site investigations expected to be undertaken by the Firm.

It should be understood by all who utilize this package that every safety hazard associated with hazardous waste site investigations cannot be covered in this manual. Safe conduct in such investigations must rely on the application of common sense, sound judgment and thorough technical analysis by each member of the project team. Guidance packages represented by this and other supplemental documents cannot be expected to resolve every field-related issue. Only a high level of safety consciousness can be expected to detect and resolve individual issues associated with safe conduct in areas known or suspected to be contaminated with hazardous materials.

Each employee should obtain and read carefully the O'Brien & Gere Safety Manual: copies are available from the personnel manager. In all cases, the employee has the option to decline to undertake the work for safety reasons, until he has performed such preliminary steps so that he can proceed with the work in a safe manner.

No phase of the work is so important, nor is any time restriction so rigid that any employee should sacrifice care or following the rules to gain a few minutes.

II. RESPONSIBILITY

The responsibility for the development and implementation of safe conduct relative to hazardous waste site investigations and remediation activities rests with each member of the project team and the supporting staff. General responsibilities for particular job classifications are outlined as follows:

Managing Engineers

Managing Engineers are responsible for the development and overall effectiveness of the safety program identified for each specific hazardous waste site investigation and/or remediation activity. This involves the planning, staffing, allocation of resources, periodic review of procedures and appropriate disciplinary action when unsafe practices are displayed.

Hazardous Materials Safety Committee

Members of the Hazardous Materials Safety Committee shall be responsible for the review of all site specific safety plans developed by the Managing Engineer. Site work shall not procede until the committee has approved the specific safety procedures and equipment prepared to be used during work completion. Additionally, the Safety Committee shall:

- advise project team members regarding safety matters
- research and distribute information regarding known hazardous conditions and required practices.
- conduct safety training for members of the project team
- monitors the specific hazardous waste site investigation safety program as required.

Project Engineer and/or Research Engineer

The principal members of any hazardous waste site investigation project team are assigned project and research engineers. It is this level of the project team which has the responsibility to see that:

- the proper equipment is available and in working order
- proper protective clothing and supplies are available and maintained
- technicians and researchers are updated relative to risks and appropriate precautionary measures
- the safety program is put into practice

- the attention of the Project Manager is immediately called to any unsafe conditions that may exist while conducting field operations

- immediate corrective action is initiated when an unsafe procedure or condition is noted

- a constructive critique relative to safety aspects of the project is provided as required.

III. GENERAL SAFETY PRECAUTIONS

Unsafe Situations

All members of the project team are directed to bring to the attention of the most readily accessible supervisor any unsafe condition, practice, or circumstance associated with or resulting from hazardous waste site investigations.

It is the responsibility of any member of the project team to take all practicable steps to eliminate or neutralize any immediate hazard to employees or the public encountered while conducting field investigations at a hazardous waste site. The Project Manager, Managing Engineer, Division Vice President and Hazardous Waste Safety Committee are to be consulted at the first opportunity. In such a situation the Project Manager, in conjunction with the Managing Engineer, Division Vice President, and Hazardous Waste Safety Committee will at the earliest point in time review those steps necessary to ensure that the investigation can be continued safely. The Project Manager may be required to implement one or more of the following steps:

- changes in procedure
- removal or neutralization of the hazard
- consultation with appropriate specialists (toxicologist, ordinance expert, etc.)
- bringing in specialists such as Explosive Ordinance Disposal units

In cases where the hazard is not immediate, the employee shall consult with the Project Manager and Managing Engineer regarding appropriate corrective measures. The application of good judgment and common sense on the part of all members of the project team is necessary when unsafe conditions are encountered. Any modifications in a sites safety plan and procedures must be approved by the Hazardous Waste Safety Committee prior to implementation.

Forbidden Practices

The following practices are expressly forbidden during operations on suspected or known hazardous waste disposal sites:

1. Smoking, eating or drinking while on the site.
2. Ignition of flammable liquids (space heaters, etc.) on the site.
3. Entry onto the site without the required protective equipment.
4. Conduct of on-site operations without sufficient personnel.

5. Entry into areas having radiation levels in excess of 10 mr/hr and explosivity readings greater than 50% LEL.

6. Movement of containers having volumes greater than 5 gallons.

7. Opening sealed vessels without full knowledge of contents and approval of supervisor.

8. Knowingly placing oneself, O'Brien & Gere staff, subcontractor's staff, the public, or the client's staff in any situation which could endanger the health and welfare of such persons.

Equipment Inspection

Prior to mobilizing to a site that involves work with potentially hazardous materials, all equipment must be inspected to assure it is in proper working order. This should be completed with a member of the Hazardous Materials Safety Committee. Equipment to be inspected shall include:

- air borne contaminant monitoring instruments
- radioactivity monitoring instruments
- respiratory protective equipment
- protective clothing

Health and Training

All employees who will engage in hazardous waste site field investigations must complete a comprehensive health examination. This examination will be paid by the employer, who will receive a written report of the results. The employee must be shown to be free of residual effects of exposure to hazardous materials and be in general good health and physical condition. The comprehensive examination is to be repeated at intervals no greater than annually for so long as the employee continues hazardous waste site investigative work.

All employees engaged in the hazardous waste site field work will receive training in basic first aid, cardio-pulmonary resuscitation and the use of protective clothing and equipment. Management is responsible for providing the necessary training at the earliest practicable time, and refresher training at appropriate times. Each Division Vice President is to identify appropriate staff for required training. The Personnel Manager is responsible for scheduling the training and identifying the appropriate update.

All staff associated with hazardous waste site field work (Division Vice Presidents, Managing Engineers, Project Managers, Project Engineers and Technicians) are required to familiarize themselves with the EPA Occupational Health and Safety Manual, 29 CFR 1910, 29 CFR 1960, and EPA Accident Reporting Procedures.

The law provides that the employer maintain for all time a complete record of every employee exposed to hazardous waste site investigations, including dates, site locations, exposure, physical problems arising and remedies taken at the time and subsequently. This is to ensure lifetime protection for such exposed employees. Diaries will be kept and become a part of job files; such records are to be filed by employees.

IV. SAFETY PROCEDURES FOR FIELD EVALUATIONS OF SITES CONTAINING HAZARDOUS MATERIALS

Introduction

The Managing Engineer together with his Project Manager is responsible for the development and implementation of the site specific safety plan. Additionally, the Managing Engineer is responsible for obtaining the review and approval of the Hazardous Materials Safety Committee.

The Project Manager ensures that all participants conduct their work in accordance with the safety plan and applicable rules. He is authorized to direct any assigned employee to leave the hazardous waste site if the employee fails to observe safety requirements or in any way creates a safety hazard.

Site Specific Safety Plan Development

A. Background Information Review

Prior to accessing a site suspected of containing hazardous materials, all available information regarding the types and quantities of potentially hazardous materials on site shall be reviewed. Particular attention should be paid 'to identified contaminants and their associated health risks. Furthermore, their modes of transportation should also be evaluated with respect to the available data; specifically these are to include:

- air
- surface water/surface runoff
- groundwater system

This information will then be used to develop the preliminary safety plan for the site and determine the diversity of the safety plan as the risks may be different for different on-site activities i.e. test drilling versus sediment sampling. The site should then be classified according to the level of protection required to perform necessary on-site activities safety. The classification system to be used shall be that used by the USEPA which ranges from Level A (highest degree of protection) to Level D (lowest degree of protection). The specific criteria used in the USEPA classification system is included in Appendix A. Information used for site classification shall be documented on Hazardous Substance Data Sheets examples of which are contained in Appendix B. The resultant Site Safety Plan shall then be developed and documented on the forms contained in Appendix C. The Hazardous Substance Data Sheets and Site Safety Plan shall be delivered to all members of the Hazardous Materials Safety Committee for their review. A site specific safety plan shall not be implemented prior to its approval by a majority of the Hazardous Materials Safety Committee members. Subsequent to committee approval, all field crew members shall be instructed as to the site specific safety plan.

B. Reconnaissance Site Description

Subsequent to the background information review, a site reconnaissance inspection shall be completed to confirm the available data and safety plan adequacy. Additionally, physical site hazards (debris, overhead clearances, equipment accessibility) should be evaluated at this time and reported on the safety plan data sheets.

A detailed discussion of those observations to be made during the reconnaissance inspection is provided in Appendix D. With specific regards to air borne contaminants, however, total organic vapor concentrations shall be monitored throughout the reconnaissance inspection. The detail of this monitoring should be sufficient to identify specific on site "hot spots". The information gathered during the organic vapor survey shall be used to either confirm or redefine respiratory protection. Rational for using total organic vapor concentrations to select respiratory protection is presented in Appendix E.

Should respiratory protection not be warranted at a specific site, chemical respirators or self contained breathing apparatus are still required to be on-site an accessible to crew members at all times.

The need for a reconnaissance inspection may be waived if earlier workers have been on site and the chemical and physical hazards of a site are well documented.

The information review and reconnaissance should also include careful examination of possible hazards to the public. Such hazards may include contamination of groundwater supplies by drilling operations, release of toxic gases, or explosion/fire. Any such hazards must be avoided or eliminated, or appropriate measures must be taken to protect the public and public property. Any indication of the presence of explosives is to be the basis for an initial investigation and appropriate followup by Army Explosive Ordinance Disposal (EOD) personnel or police explosives unit. In the event of a situation dangerous to public safety, field personnel are to immediately notify the responsible managing engineer and civil authorities.

Before entry on a suspect or known hazardous waste site, all investigative personnel must know the locations and emergency telephone numbers for the nearest medical facility, ambulance service, fire department, police department, poison control centers and EPA Office contact.

Clean Area

During operations on a suspect or known hazardous waste site, a "clean" area must be established outside the area of suspected contamination. At least one backup team member will remain in this area to:

1. Assist in emergency removal of team members from the hazardous waste site in the event of accident or injury. The backup must have readily available protective clothing, breathing apparatus and first aid equipment.

2. Assist in moving equipment, samples, and supplies.

3. Provide communication to emergency units.

4. Assist in decontamination or removal of contaminated clothing from the individuals emerging from the contaminated area.

5. As appropriate, prevent entry of unauthorized persons to the hazardous waste site while operations are underway.

6. Provide other assistance as necessary, but with the primary objective of facilitating safe transfer of personnel and equipment to and from the contaminated area.

Sampling Equipment

As a general rule, sampling equipment used on hazardous waste sites should be disposable. Dippers, scoops and similar devices for solids samples should be buried onsite, or placed in plastic bags for disposal or later decontamination. Liquid samples from barrels or tanks should be withdrawn in inert tubing, such as glass, and the tubing should then be broken and abandoned within the barrel or tank. If incineration or recycling of barrel contents is contemplated, the tubing may be disposed of in other suitable containers, or buried on the site. Particular care should be given to the application of the Composite Liquid Waste Sampler (Coliwasa) because it is difficult to decontaminate under field conditions.

Clothing

Protective clothing must be worn by all assigned personnel while on a suspected or confirmed hazardous waste site, until sufficient data has been acquired to enable the Project Manager to make an informed judgment regarding the need. Project Managers must weigh the fact that fatigue and alertness on the part of the team members is a significant safety factor. Protective clothing is cumbersome, hastens the on-set of fatigue, and limits stay-time. In the absence of clear indications that work can proceed safely without protective clothing, required items include chemical-resistant pants and jacket, rubber boots, protective gloves, hard hat or head cover, face shield or chemical safety glasses.

Disposable and reusable clothing is available, and each has advantages and disadvantages. The presently available disposable clothing is fragile, easily torn, and especially vulnerable during cold weather. The "bootees" that are furnished with this clothing are highly vulnerable and are of limited value on rough ground or for walking through snagging objects. Reusable clothing is available in much sturdier fabric and is generally preferred. The disadvantage is the necessity for decontamination on-site, or careful packaging, shipment and later decontamination. The reusable suits are worn with heavy rubber slip-on boots, which are easily decontaminated on-site with reagent solution.

Full decontamination of reusable suits is accomplished in two steps. The first step is performed on-site using a reagent solution selected beforehand in consultation with Chemistry and Biology personnel, based on limited knowledge of chemical and biological hazards on the site at that time. After cleaning protective clothing is turned inside out, if feasible, and

sealed in plastic bags for return shipment. The second decontamination step is taken later, after enough of the sample has been laboratory-analyzed to determine what decontamination reagents are most suitable for each case. This second cleaning is then performed by personnel wearing disposable safety clothing. Waste decontamination solution from the second step should be treated as hazardous waste and disposed of according to appropriate regulations.

SCBA/Respirators

Self-contained breathing apparatus (SCBA) must be worn on-site when:

- still air conditions prevail
- containers of unknown or known hazardous materials are being opened
- in enclosed spaces, such as unventilated buildings or rooms
- under any circumstances where free-flow of air uncontaminated by toxics is in doubt.

In cases where the Project Manager has determined that on-site work may proceed without use of SCBA, participating personnel must carry respirators having organic vapor protection cartridges, or combination cartridges. An oxygen meter should be used to determine that at least 19.5% oxygen is present in the area where respirators are to be used. Respirators should be donned immediately upon experiencing breathing difficulty, dizziness or other distress, strong taste or smell, or mere judgment that precaution is in order. Once respirators have been donned, team members should withdraw from the site pending a decision by the Project Manager regarding continued operations. Cartridge respirators should not be relied upon for protection from organic vapors for extended periods.

Remember:

1. Respirator cartridges for organic vapors function as adsorbants. Once adsorptive capacity is reached, the cartridge no longer functions.
2. Cartridge respirators do not supply oxygen. They are of no use in oxygen deficient atmospheres.

Sampling Procedures

Sampling methods are described in the "Waste Disposal Site Hazard Assessment Manual." As indicated under "Field Sampling" above, disposable sampling equipment should be used wherever possible. The guiding safety principle is to prevent exposure of personnel doing sampling, packaging, shipping, analysis, and to prevent exposure of others to spilled or residual waste materials.

Hazardous waste site sample volume should be the smallest consistent with analytical requirements. Sample containers must be cleaned and free of

spilled or residual waste material on the exterior of the container, prior to shipment (see Chapter V - "Shipping of Hazardous Waste Site Samples").

Leaving the Site

Procedures for leaving the suspect contaminated area must be planned before entry. Provision must be made for: decontamination and safe packing of protective clothing; burial or safe packing of disposable gear; handling of samples and preparation of samples for shipment; transfer of equipment, gear, and samples from the "contaminated" area to the "clean" area; etc. Sequences will depend on several variables -- such as SCBA inside or outside of protective clothing -- but must be worked out in advance. A detailed discussion of acceptable decontamination procedures at different protection levels is contained in Appendix F.

Training

Field team leaders in hazardous waste site investigations must be provided hands-on training on simulated sites in order to achieve competence in the safety and operational aspects. Preparation for on-site investigations must include detailed briefings, particularly for inexperienced personnel. The requirement for planning and carefully-thought-out sequences must be stressed.

V. PACKAGING, MARKING, LABELING AND SHIPPING OF HAZARDOUS WASTE SITE SAMPLES

General Provisions

These procedures apply to samples collected from a hazardous waste site but which in the judgment of the Project Leader cannot be considered to be "environmental" samples.

- Unanalyzed hazardous waste site samples may not be fixed with any preservative or preserved with ice or dry ice.

- If a material specifically identified in the Department of Transportation (DOT) Hazardous Material Table (49 CFR 172.101) is known to be contained in the hazardous waste site sample, that sample should be transported as prescribed in the table. Samples that are judged to be environmental samples may be shipped according to letters of understanding granted OB&G by EPA, DOT or NYSDEC. Other specific exemptions may also apply.

- Unanalyzed hazardous waste site samples may be transported by rented or common carrier truck, bus, railroad, and by Federal Express Corporation (air cargo); but they may not be transported by any other common carrier air transport, even "cargo only" aircraft. Those samples taken from closed drums or tanks, however, should not ordinarily be transported by Federal Express.

Containers (drums, tanks etc.) should be sampled only when necessary to meet enforcement or cleanup requirements. Opening drums or other sealed containers may be hazardous to field personnel unless proper safety procedures are followed. Gases can be released, or pressurized liquids can be expelled. A drum should not be moved or opened unless it can be ascertained beyond reasonable doubt that the drum is structurally sound. Drums standing on end, with bung up, should be opened by pneumatic impact wrench, operated from a remote site. Drums on sides may be opened similarly if it is possible to safely rotate the drum so that the bung is high. If the bung can be removed, sampling contained liquids may be safely accomplished by glass tube, which is then broken and discarded with the barrel. A barrel that has a badly rusted bung, or that cannot be sampled as above, may be safely sample sampled with a hydraulic penetrating device (Figure 3) operated remotely. The device is then abandoned in place, and disassembled to prevent further withdrawal of liquids. Sealed or closed tanks should be opened remotely, using ropes to lift hatches, etc.

In general, metal sample containers should not be used on hazardous waste site investigations; if used, they must be grounded, preferably to the drum or tank being sampled, while sample transfer is accomplished.

In all cases of entry into closed containers, the local fire department should be asked to stand by. In any case wherein presence of explosives is suspected or known, Army EOD or police bomb squads should be requested to remove or neutralize such materials. In no event may

O'Brien & Gere employees or subcontractors handle explosives encountered on dump sites.

Subsurface sampling of hazardous waste sites can also create hazards to employees and the public, unless adequate safety precautions are followed. Biodegradation of refuse in dumps produces methane and other explosive gases. The escaping gases may be ignited by drill rigs or other ignition sources. Drilling into dump sites may cause discarded incompatibles to be mixed and thereby create reactive mixtures. Dump sites where leachate plumes are contained on impervious strata may be interconnected with producing aquifers if drilling is not planned according to competent groundwater technology and data.

Drilling in hazardous waste site investigations should be confined to the periphery of dump sites, with the objective of characterizing the leachate that may be moving away from the site. If subsurface sampling of dump sites is necessary, excavation must be accomplished by hand, and with spark-free equipment.

All drilling associated with hazardous waste site investigations must be accomplished under the responsible supervision of a competent geohydrologist, groundwater geologist, geological engineer, or a person similarly qualified by experience. Drilling must be preceded by sweeps with metal detectors, and drilling must be limited to areas where the presence of buried drums or tanks is not indicated. Test holes must be cased or plugged when the investigation is completed.

Ambient air sampling on hazardous waste sites must be accomplished with spark-free equipment if explosive vapors are present (most hi-vol samplers are spark sources).

Samples from hazardous waste sites must not be preserved, or "fixed," by the addition of chemicals (see "Waste Disposal Site Hazard Assessment Manual" regarding cooling of samples in ice chests or refrigerators).

Preliminary Steps

- Conduct a radiation measurement if radiation contamination is suspected to eliminate the possibility of a sample being radioactive.

- Place a sufficient quantity of sample in glass and/or polyethylene containers to determine whether sample material will react with or substantially reduce the effectiveness of the container (this should be done at time of sampling).

- Pack samples according to the "Packaging, Marking and Labeling Requirements for Unanalyzed Hazardous Waste Samples taken from Closed Drums," as discussed below.

Packaging, Marking and Labeling Requirements for Unanalyzed Hazardous Waste Site Samples, Excluding Drum Samples

1. Collect sample in a 16-ounce* or smaller glass or polyethylene container with nonmetallic, teflon-lined screw cap. Allow sufficient ullage

(approximately 10% by volume) so container is not liquid full at 130° F. If collecting a solid material, the container plus contents shall not exceed 1 pound net weight. If sampling for volatile organic analysis (VOA), fill special VOA container to septum but use special cap to accomplish an air space within the container.

2. Attach properly completed sample identification tag to sample container.

3. Seal sample container and place in 2-mil-thick (or thicker) polyethylene bag, one sample per bag. (Tags should be positioned to enable them to be read through bag.)

4. Place sealed bag inside a metal can with incombustible, absorbent cushioning material (e.g., vermiculite or earth) to prevent breakage, one bag per can. Pressure-close the can and use clips, tape or other positive means to hold the lid securely, tightly and effectively.

5. Mark and label this container as indicated in No. 8 below.

6. Mark and label the outside container and complete shipping papers as described in No. 8 below.

Marking and Labeling

Use abbreviations only where specified. Place the following information on a metal can (or bottle), either hand printed or in label form: laboratory name and address and "Flammable Liquid, n.o.s.", (if not liquid, write "Flammable Solid, n.o.s."). Place the following labels on the outside of the can (or bottle), next to one another and near the "Flammable liquid, n.o.s." marking:

"Cargo Aircraft Only"; "Flammable Liquid"; (if not liquid, "Flammable Solid" and "Dangerous When Wet").

Note: If the cans are placed in an exterior container, both that container and inside cans must have the same markings and labels as above. "Laboratory Samples" and "THIS SIDE UP" or "THIS END UP" should also be marked on the top of the outside container, and upward pointing arrows should be placed on all four sides of the exterior container.

Shipping Papers

Complete the carrier-provided bill of lading and sign the certification statement (if carrier does not provide, use standard industry form) with the following information in the order listed. One form may be used for more than one exterior container, for example:

"Flammable Liquid, n.o.s." (or "Flammable Solid, n.o.s.", as appropriate); "Cargo Aircraft Only"; "Limited Quantity" or "Ltd. Qty."; "Laboratory Samples"; "Net Weight _____" or "Net Volume _____" (of hazardous contents), by item, if more than one metal can is inside an exterior container. The net weight or net volume must be placed just before or just after the

"Flammable Liquid, n.o.s." or "Flammable Solid, n.o.s." description.

A Chain-of-Custody Record form (Figure 2) should also be properly executed, and included in the exterior container.

Packaging, Marking and Labeling Requirements for Unanalyzed Hazardous-Waste Site Samples Taken from Closed Drums

1. All samples from closed drums do not necessarily have to be shipped as below. The Project Coordinator must make a judgement as to the hazard class of samples gathered. This packaging, marking, labeling and shipping method provides a worst-case procedure for materials classed as "Poison A" (49 CFR 173.328).

2. These samples may not be transported by Federal Express Corporation (air cargo) or other common carrier aircraft, or by rental, non-government aircraft. (Samples may be shipped by ground transport or government aircraft.)

3. Collect sample in a polyethylene or glass container which is of an outer diameter narrower than the valve hole on a DOT Spec. 3A1800 or 3AA1800 metal cylinder. Fill sample container allowing sufficient ullage (approximately 10% by volume) so it will not be liquid- full at 130°F. If sampling for volatile organic analysis, fill special VOA container to septum, but use special cap to achieve an air space within the container. Seal sample container.

4. Attach properly completed Sample Identification Tag (Figure 4) to sample container.

5. With a string or flexible wire attached to the neck of the sample container, lower it into a metal cylinder which has been partially filled with incombustible, absorbent, loose packaging material (vermiculite or earth). Allow sufficient cushioning material between the bottom and sides of the container and the metal cylinder to prevent breakage. After the cylinder is filled with cushioning material, drop the ends of the string or wire into the cylinder valve hole. Only one sample container may be placed in a metal cylinder.

6. Replace valve and valve protector on metal cylinder.

7. Mark and label cylinder as described in No. 10 below.

8. One or more cylinders may be placed in a strong outside container.

9. Mark and label outside container and complete shipping papers as described in No. 10 below.

Marking and Labeling

Use abbreviations only where specified. Place the following information on the side of the cylinder, or on a tag wired to the cylinder valve protector, either hand-printed or in label form.

10. Unless samples are driven to the laboratory, an OB&G employee will accompany shipping containers to the transport carrier and, if required, open outside container(s) for freight inspection.

"Poisonous Liquid or Gas, n.o.s."; laboratory name and address.*

Place the following label on the cylinder: "Poisonous Gas". ("Poisonous Liquid" label not acceptable here, even if liquid.)

Note: If the metal cylinders are placed in an outside container, both the container and cylinders inside must have the same markings and labels as above. In addition, "Laboratory Sample", and "Inside Packages Comply With Prescribed Specifications" should be marked on the top of the outside container. "THIS SIDE UP" marking should be placed on the outside container and upward pointing arrows on four sides.

Shipping Papers: Complete the shipper-provided bill of lading and sign the certification statement (if carrier does not provide, use standard industry form) with the following information in the order listed. One form may be used for more than one exterior container; use abbreviations only as specified:

"Poisonous Liquid, n.o.s."; "Limited Quantity" or "Ltd. Qty."; "Laboratory Samples"; "Net Weight _____" or "Net Volume _____" (of hazardous contents), by cylinder, if more than one cylinder is inside an exterior container. The net weight or net volume must be placed just before or just after the "Poisonous Liquid, n.o.s." marking.

A Chain-of-Custody Record for (Figure 2) should also be properly executed and included in the container.

VI. SUMMARY

Human safety should be the first priority in activities related to Hazardous Waste Site Investigations. In all cases, the employee has the option to decline to undertake the work for safety reasons, until he has performed such preliminary steps so that he can proceed with the work in a safe manner.

When hauling samples in O'Brien & Gere vehicles, drivers should be especially cautions. Whether the vehicle has a total collision (self-only) or impact with another vehicle, specific actions can be taken. Consideration should be taken concerning the samples themselves should they be spilled or catch fire. Drivers in all cases should look to self preservation, first and foremost. Subsequently, police should be notified immediately; the area should be isolated; and the home office should be contacted for further instructions.

APPENDIX A

LEVELS OF PROTECTION

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LEVELS OF PROTECTION

I. INTRODUCTION

Personnel must wear protective equipment when response activities involve known or suspected atmospheric contamination, when vapors, gases, or particulates may be generated, or when direct contact with skin-affecting substances may occur. Respirators can protect lungs, gastrointestinal tract, and eyes against air toxicants. Chemical-resistant clothing can protect the skin from contact with skin-destructive and -absorbable chemicals. Good personal hygiene limits or prevents ingestion of material.

Equipment to protect the body against contact with known or anticipated chemical hazards has been divided into four categories according to the degree of protection afforded:

- Level A: Should be worn when the highest level of respiratory, skin, and eye protection is needed.

- Level B: Should be selected when the highest level of respiratory protection is needed, but a lesser level of skin protection. Level B protection is the minimum level recommended on initial site entries until the hazards have been further defined by on-site studies and appropriate personnel protection utilized.

- Level C: Should be selected when the type(s) of airborne subtance(s) is known, the concentration(s) is measured, and the criteria for using air-purifying respirators are met.

- Level D: Should not be worn on any site with respiratory or skin hazards. Is primarily a work uniform providing minimal protection.

The Level of Protection selected should be based primarily on:

- Type(s) and measured concentration(s) of the chemical substance(s) in the ambient atmosphere and its toxicity.

- Potential or measured exposure to substances in air, splashes of liquids, or other direct contact with material due to work being performed.

In situations where the type(s) of chemical(s), concentration(s), and possibilities of contact are not known, the appropriate Level of Protection must be selected based on professional experience and judgment until the hazards can be better characterized.

While personnel protective equipment reduces the potential for contact with harmful substances, ensuring the health and safety of response personnel requires, in addition, safe work practices, decontamination, site entry

protocols, and other safety considerations. Together, these protocols establish a combined approach for reducing potential harm to workers.

II. LEVELS OF PROTECTION

A. Level A Protection

1. Personnel protective equipment

- Pressure-demand, self-contained breathing apparatus, approved by the Mine Safety and Health Administration (MSHA) and National Institute of Occupational Safety and Health (NIOSH).
- Fully encapsulating chemical-resistant suit
- Coveralls*
- Long cotton underwear*
- Gloves (outer), chemical-resistant
- Gloves (inner), chemical-resistant
- Boots, chemical-resistant, steel toe and shank. (Depending on suit construction, worn over or under suit boot)
- Hard hat* (under suit)
- Disposable protective suit, gloves, and boots* (Worn over fully encapsulating suit)
- 2-Way radio communications (intrinsically safe)

2. Criteria for selection

Meeting any of these criteria warrants use of Level A Protection:

- The chemical substance(s) has been identified and requires the highest level of protection for skin, eyes, and the respiratory system based on:
  - -- measured (or potential for) high concentration(s) of atmospheric vapors, gases, or particualtes

    or

  - -- site operations and work functions involving high potential for splash, immersion, or exposure to unexpected vapors, gases, or particulates.

*Optional

- Extremely hazardous substances (for example: dioxin, cyanide compounds, concentrated pesticides, Department of Transportation Poison "A" materials, suspected carcinogens, and infectious substances) are known or suspected to be present, and skin contact is possible.

- The potential exists for contact with substances that destroy skin.

- Operations must be conducted in confined, poorly ventilated areas until the absence of hazards requiring Level A protection is demonstrated.

- Total atmospheric readings on the Century OVA System, HNU Photoionizer, and similar instruments indicate 500-1,000 ppm of unidentified substances. (See Appendixes I and II.)

3. Guidance on selection criteria

The fully encapsulating suit provides the highest degree of protection to skin, eyes, and respiratory system if the suit material is resistant to the chemical(s) of concern during the time the suit is worn and/or at the measured or anticipated concentrations. While Level A provides maximum protection, the suit material may be rapidly permeated and penetrated by certain chemicals from extremely high air concentrations, splashes, or immersion of boots or gloves in concentrated liquids or sludges. These limitations should be recognized when specifying the type of chemical-resistant garment. Whenever possible, the suit material should be matched with the substance it is used to protect against.

The use of Level A protection and other chemical-resistant clothing requires evaluating the problems of physical stress, in particular heat stress associated with the wearing of impermeable protective clothing. Response personnel must be carefully monitored for physical tolerance and recovery.

Protective equipment being heavy and cumbersome, decreases dexterity, agility, visual acuity, etc., and so increases the probability of accidents. This probability decreases as less protective equipment is required. Thus, increased probability of accidents should be considered when selecting a Level of Protection.

Many toxic substances are difficult to detect or measure in the field. When such substances (especially those readily absorbed by or destructive to the skin) are known or suspected to be present and personnel contact is unavoidable, Level A protection should be worn until more accurate information can be obtained.

B. Level B Protection

1. Personal protective equipment

- Pressure-demand, self-contained breathing apparatus (MSHA/NIOSH approved)

- Chemical-resistant clothing (overalls and long-sleeved jacket; coveralls; hooded, one or two-piece chemical-splash suit; disposable chemical-resistant coveralls)

- Coveralls*

- Gloves (outer), chemical-resistant

- Gloves (inner), chemical-resistant

- Boots (outer), chemical-resistant, steel toe and shank

- Boots (outer), chemical-resistant (disposable)*

- Hard hat (face shield*)

- 2-Way radio communications (intrinsically safe)

2. Criteria for selection

Meeting any one of these criteria warrants use of Level B protection:

- The type(s) and atmospheric concentration(s) of toxic substances have been identified and require the highest level of respiratory protection, but a lower level of skin and eye protection. These would be atmospheres:

  -- with concentrations Immediately Dangerous to Life and Health (IDLH)

  or

  -- exceeding limits of protection afforded by a full-face, air-purifying mask

  or

  -- containing substances for which air-purifying canisters do not exist or have low removal efficiency

  or

  -- containing substances requiring air-supplied equipment, but substances and/or concentraions do not respresent a serious skin hazard.

- The atmosphere contains less than 19.5% oxygen.

- Site operations make it highly unlikely that the small, unprotected area of the head or neck will be contacted by splashes of extremely hazardous substances.

*Optional

- Total atmospheric concentrations of unidentified vapors or gases range from 5 ppm to 500 ppm on instruments such as the Century OVA System or HNU Photoionizer, and vapors are not suspected of containing high levels of chemicals toxic to skin. (See Appendixes I and II.)

3. Guidance on selection criteria

Level B equipment provides a high level of protection to the respiratory tract, but a somewhat lower level of protection to skin. The chemical-resistant clothing required in Level B is available in a wide variety of styles, materials, construction detail, permeability, etc. These factors all affect the degree of protection afforded. Therefore, a specialist should select the most effective chemical-resistant clothing (and fully encapsulating suit) based on the known or anticipated hazards and/or job function.

Generally, if a self-contained breathing apparatus is required, Level B clothing rather than a Level A fully encapsulating suit is selected, based on the protection needed against known or anticipated substances affecting the skin. Level B skin protection is selected by:

- Comparing the concentrations of known or identified substances in air with skin toxicity data.

- Determining the presence of substances that are destructive to and/or readily absorbed through the skin by liquid splashes, unexpected high levels of gases or particulates, or other means of direct contact.

- Assessing the effect of the substance (at its measured air concentrations or splash potential) on the small area of the head and neck unprotected by chemical- resistant clothing.

For initial site entry and reconnaissance at an open site, approaching whenever possible from the upwind direction, Level B protection (with good quality, hooded, chemical-resistant clothing) should protect response personnel, providing the conditions described in selecting Level A are known or judged to be absent. For continuous operations, the aforementioned criteria must be evaluated.

At 500 pm total vapors/gases, upgrading to Level A protection may be advisable. A major factor for re-evaluation is the presence of vapors, gases, or particulates requiring a higher degree of skin protection.

Level C Protection

1. Personal protective equipment

   - Full-face, air-purifying, canister-equipped respirator (MSHA/NIOSH approved)

   - Chemical-resistant clothing (coveralls; hooded, two-piece chemical

splash suit; chemical-resistant hood and apron; disposable chemical-resistant coveralls)

- Coveralls*
- Gloves (outer), chemical-resistant
- Gloves (inner), chemical-resistant*
- Boots (outer), chemical-resistant, steel toe and shank*
- Boots (outer), chemical-resistant (disposable)*
- Hard hat (face shield*)
- Escape mask*
- 2-Way radio communications (intrinsically safe)

2. Criteria for selection

Meeting all of these criteria permits use of Level C protection:

- Measured air concentrations of identified substances will be reduced by the respirator to at or below the substance's exposure limit, and the concentration is within the service limit of the canister.
- Atmospheric contaminant concentrations do not exceed IDLH levels.
- Atmospheric contaminants, liquid splashes, or other direct contact will not adversely affect the small area of skin left unprotected by chemical-resistant clothing.
- Job functions have been determined not to require self-contained breathing apparatus.
- Total vapor readings register between background and 5 ppm above background on instruments such as the HNU Photoionizer and Century OVA System. (See Appendixes I and II.)
- Air will be monitored periodically.

3. Guidance on selection criteria

Level C protection is distinguished from Level B by the equipment used to protect the respiratory system, assuming the same type of chemical-resistant clothing is used. The main selection criterion for Level C is that conditions permit wearing air-purifying devices.

The air-purifying device must be a full-face mask (MSHA/NIOSH approved) equipped with a canister suspended from the chin or on a harness. Canisters

*Optional

must be able to remove the substances encountered. Quarter- or half-masks or cheek-cartridge full-face masks should be used only with the approval of a qualified individual.

In addition, a full-face, air-purifying mask can be used only if:

- Oxygen content of the atmosphere is at least 19.5% by volume.
- Substance(s) is identified and its concentration(s) measured.
- Substance(s) has adequate warning properties.
- Individual passes a qualitative fit-test for the mask.
- Appropriate cartridge/canister is used, and its service limit concentration is not exceeded.

An air monitoring program is part of all response operations when atmospheric contamination is known or suspected. It is particularly important that the air be monitored throroughly when personnel are wearing air-purifying respirators (Level C). Continual surveillance using direct-reading instruments and air sampling is needed to detect any changes in air quality necessitating a higher level of respiratory protection. See Part 8 for guidance on air monitoring.

Total unidentified vapor/gas concentrations of 5 ppm above background require Level B protection. Only a qualified individual should select Level C (air-purifying respirators) protection for continual use in an unidentified vapor/gas concentration of background to 5 ppm above background.

D. Level D Protection

1. Personal protective equipment

   - Coveralls

   - Gloves*

   - Boots/shoes, leather or chemical-resistant, steel toe and shank

   - Boots (outer), chemical-resistant (disposable)*

   - Safety glasses or chemical splash goggles*

   - Hard hat (face shield)*

   - Escape mask*

2. Criteria for selection

   Meeting any of these criteria allows use of Level D protection:

   No hazardous air pollutants have been measured.

Work functions preclude splashes, immersion, or potential for unexpected inhalation of any chemicals.

3. Guidance on selection criteria

Level D protection is primarily a work uniform. It can be worn in areas where: 1) only boots can be contaminated, or 2) there are no inhalable toxic substances.

III. PROTECTION IN UNKNOWN ENVIRONMENTS

In all site operations, selecting the appropriate personnel protection equipment is one of the first steps in reducing the potential for adverse health effects. Until the hazardous conditions presented by an environmental incident can be identified and personnel safety measures commensurate with the hazards - real or potential - instituted, preliminary measures will have to be based on applying experience, judgment, and professional knowledge to the particular incident at hand. Lack of knowledge concerning the hazards that could be encountered precludes selecting protective equipment by comparing environmental concentrations of known toxicants against protection afforded by each type of equipment.

One of the first considerations in evaluating the risk of an unknown environment is to measure immediate atmospheric hazards such as the concentrations (or potential concentrations) of vapors, gases, and particulates; oxygen content of the air; explosive potential; and, to a lesser degree, the possibility of radiation exposure. In addition to air measurements, visual observation and/or evaluation of existing data can help determine the degree of risk from other materials that are explosive, have a high fire potential, are extremely toxic, or exhibit other hazardous characteristics that cannot be monitored by field instruments.

Total vapor/gas concentration as indicated by instruments such as the Century OVA System or the HNU Photoionizer is a useful adjunct to professional judgment in selecting the Level of Protection to be worn in an unknown environment. It should not be the sole criterion, but should be considered with all other available information. Total vapor/gas concentration should be applied only by qualified persons thoroughly familiar with the information contained in Appendixes I and II.

The initial on-site survey and reconnaissance, which may consist of more than one entry, is to characterize the immediate hazards and, based on these findings, establish preliminary safety requirements. As data are obtained from the initial survey, the Level of Protection and other safety procedures are adjusted. Initial data also provide information on which to base further monitoring and sampling. No method can select a Level of Protection in all unknown environments. Each situation must be examined individually. Some general approaches can be given, however, for judging the situation and determining the Level of Protection required.

- Entering cloud of chlorine released in a railroad accidnent.

- Handling and moving drums suspected and/or known to contain substances that were skin destructive or absorbable.

- Responding to accidents involving cyanide, arsenic, or undiluted pesticides.

C. Level B

While Level B protection does not afford the maximum skin (and eye) protection as does a fully encapsulating suit, a good quality, hooded, chemical-resistant, one-or-two-piece garment, with taped joints, provides a reasonably high degree of protection. At most abandoned hazardous waste sites, ambient atmospheric gas/vapor levels have not approached concentrations sufficiently high to warrant maximum protection. In all but a few circumstances, Level B should provide the protection needed for initial entry. Subsequent operations require a re-evaluation of Level B based on the probability of being splashed by chemicals, their effect on the skin, or the presence of hard-to-detect air contaminants.

IV. ADDITIONAL CONSIDERATIONS

In addition to the topics previously addressed, there are other factors which should be considered in selecting the appropriate Level of Protection.

A. Protective Clothing

No adequate criteria are available, similar to the respiratory protection decision-logic, for selecting protective clothing. A concentration of a known substance in the air approaching a TLV or permissible exposure limit for the skin does not automatically warrant a fully encapsulating suit. A hooded, high quality, chemical-resistant suit may provide adequate protection. The selection of Level A over Level B is a judgment that should be made by a qualified individual considering the following factors:

- Effect of the material on skin:

  -- highly hazardous substances are those that are easily absorbed through the skin, causing systemic effects, or that cause severe skin destruction. Liquids are generally more hazardous than vapors/gases and particulates.

  -- less hazardous substances are those that are not easily absorbed through the skin, causing systemic effects, or that cause severe skin destruction

A. Level C

Level C protection (full-face, air-purifying respirator) should be worn routinely in an atmosphere only after the type(s) of air contaminant(s) is identified and concentrations measured. To permit flexibility in prescribing a Level of Protection at certain environmental incidents, a specialist could consider air-purifying respirators for use in unidentified vapor/gas concentrations of a few parts per million. The guideline of total vapor/gas concentration of background to 5 ppm above background should not be the sole criterion for selecting Level C. Since the individual contributors may never be completely identified, a decision on continuous wearing of Level C must be made, after assessing all safety considerations, including:

- The presence of (or potential for) organic or inorganic vapors/gases against which a canister is ineffective or has a short service life.
- The known (or suspected) presence in air of substances with low TLV or IDLH levels.
- The presence of particulates in air.
- The errors associated with both the instruments and monitoring procedures used.
- The presence of (or potential for) substances in air which do not elicit a response on the instrument(s) used.
- The potential for higher concentrations in the ambient atmosphere or in the air adjacent to specific site operations.

The continuous use of air-purifying respirators (Level C) should be based on the identification of the substances contributing to the total vapor/gas concentration and the application of published criteria for the routine use of air-purifying devices. Unidentified ambient concentrations of organic/vapors or gases in air approaching or exceeding 5 ppm above background require Level B protection.

Individuals without appropriate training and/or experience should be discouraged from modifying upward the recommended total vapor/gas concentration guideline and associated Levels of Protection.

B. Level A

Level A should be worn when maximum protection is needed against substances that could damage the surface of the skin and/or be absorbed through the skin. Since Level A requires the use of a self-contained breathing apparatus, the eyes and respiratory system are also protected. For initial site entry, skin toxicants would exist primarily as vapors, gases, or particulates in air, with a lesser

possibility of splash. Continuous operations at an abandoned waste site, for instance, may require Level A due to working with and around severe skin toxicants.

Until air monitoring data are available to assist in the selection of the appropriate Level of Protection, the use of Level A for initial site entries may have to be based on indirect evidence of the potential for atmospheric contamination or direct skin contact.

Considerations that may require Level A protection include:

- Confined spaces: Enclosed, confined, or poorly ventilated areas are conducive to buildup in air of toxic vapors, gases, or particulates. (Explosive or oxygen-deficient atmospheres also are more probable in confined spaces.) Low-lying outdoor areas - ravines, ditches, and gulleys - tend to accumulate any heavier-than-air vapors or gases present.

- Suspected/known toxic substances: Various substances may be known or suspected to be involved in an incident, but there are no field instruments available to detect or quantify air concentrations. In these cases, media samples must be analyzed in the laboratory. Until these substances are identified and levels measured, maximum protection may be necessary.

- Visible emissions: Visible emissions from leaking containers or railroad/vehicular tank cars, as well as smoke from chemical fires, indicate high potential for concentration of substances that could be extreme respiratory or skin hazards.

- Job functions: Initial site entries are generally walk-throughs in which instruments and/or visual observations provide a preliminary characterization of the hazards. Subsequent entries are to conduct the many activities needed to reduce the environmental impact of those hazards. Levels of Protection for later operations are based not only on data obtained from the initial and subsequent environmental monitoring, but also on the probability of contamination. Maximum protection (Level A) should be worn when:

  -- there is a high probability for exposure to high concentrations of vapors, gases, or particulates.

  -- substances could splash.

  -- substances are known or suspected of being extremely toxic directly to the skin or by being absorbed.

Examples of situations where Level A has been worn are:

- Excavating of soil suspected of being contaminated with dioxin.

APPENDIX B

HAZARDOUS SUBSTANCE DATA SHEET

HAZARDOUS SUBSTANCE DATA SHEET

NAME OF SUBSTANCE:

COMMON: _____ CHEMICAL: _____

I. PHYSICAL/CHEMICAL PROPERTIES

			SOURCE
Normal physical state:	_____ Gas	_____ Liquid	_____ Solid
Molecular weight			
Density		gm/ml	
Specific gravity	@	°F/°C	
Solubility: (water)	@	°F/°C	
Solubility: _____	@	°F/°C	
Boiling point		°F/°C	
Melting point		°F/°C	
Vapor pressure	mmHg	°F/°C	
Vapor density	@	°F/°C	
Flash point		°F/°C	
Autoignition point		°F/°C	
Other: _____		°F/°C	
_____			

II. HAZARDOUS CHARACTERISTICS

TOXICOLOGICAL	HAZARD		CONCENTRATIONS	SOURCE
Inhalation	Yes	No		
Ingestion	Yes	No		
Skin/eye absorpt.	Yes	No		
Skin/eye contact	Yes	No		
Carcinogenicity	Yes	No		
Teratogenicity	Yes	No		
Mutagenicity	Yes	No		
Aquatic	Yes	No		
Other: _____	Yes	No		

FIRE			CONCENTRATIONS	SOURCE
Combustibility	Yes	No		
Toxic byproducts	Yes	No		
_____				
_____				

EXPLOSIVENESS				
	Yes	No		
LEL				
UEL				

	HAZARD	CONCENTRATIONS	SOURCE
REACTIVITY	Yes No		
_____		_____	_____
_____		_____	_____
CORROSIVENESS			
pH _____	Yes No	_____	_____
Neutralizing agent:			
_____		_____	_____
_____		_____	_____
RADIOACTIVE	Yes No	EXPOSURE RATE	SOURCE
Alpha radiation		_____	_____
Beta radiation		_____	_____
Gamma radiation		_____	_____
Natural (background) radiation		_____	

III. INCIDENT RELATED:

Quantity involved _____

Information released on _____

_____

Monitoring/sampling recommended _____

_____

Public _____

_____

Environment _____

_____

Worker _____

_____

EMERGENCY HAND SIGNALS

1.	Hand gripping throat:	Out of air, can't breath.
2.	Grip partner's wrist or place both hands around waist:	Leave area immediately, no debate!
3.	Hands on top of head:	Need assistance.
4.	Thumbs up:	OK, I'm all right, I understand.
5.	Thumbs down:	No, negative.

APPENDIX C

SITE/AREA SAFETY PLAN

SITE/AREA SAFETY PLAN

(use last page if additional space is necessary)

GENERAL

DATES PLAN IN USE: _____ DATE PREPARED: _____

PREPARED BY: _____ SITE/AREA NAME: _____

LOCATION: _____

EXISTING INFORMATION FOR SITE: DETAILED ____ PRELIMINARY ____ SKETCHY ____ NONE ____

HAZARDOUS MATERIAL FORM: GAS ____ LIQUID ____ SLUDGE ____ SOLID ____

CONTAINMENT: DRUM ____ PIT ____ POND ____ LAGOON ____ TANK ____ SOILS ____ DEBRIS ____

OTHER _____ CONDITION _____

CHARACTERISTICS: CORROSIVE ____ IGNITABLE ____ RADIOACTIVE ____ VOLATILE ____ TOXIC ____

REACTIVE ____ OTHER _____ UNKNOWN ____

SITE/AREA SPECIFICS

HIGH HAZARD MATERIAL:

COMPOUND	ANTICIPATED CONCENTRATION	WARNING PROPERTIES

SITE HISTORY: TOPOGRAPHY _____

STATUS: OPEN ____ CLOSED ____ LIMITED ACCESS ____ UNKNOWN ____

HISTORY (regulatory action, local complaints, injuries, site controls):

_____

_____

UNUSUAL FEATURES (control feature integrity,utilities, obstacles):

_____

_____

HAZARD ASSESSMENT

EVALUATION OF EXPECTED HAZARD (work assignments, operational considerations, routes of exposure, health effects, material stability):

_____

_____

_____

_____

_____

OPERATIONAL PROCEDURES

SITE COMMAND AND CONTROL (include sketch or map as appropriate):

PERIMETER CONTROL _____

STAGING AREA _____

EQUIPMENT REQUIREMENTS _____

PERSONNEL PROTECTION

GENERAL LEVEL OF PROTECTION REQUIRED: A ____ B ____ C ____ D ____

MODIFICATION OR SPECIALIZED EQUIPMENT: _____

_____

DETECTION EQUIPMENT (survey meters, dosimeters):

_____

_____

COMMUNICATIONS (type, range, frequencies, alternates, hand signals):

_____

_____

AUTHORIZED TEAM PERSONNEL

NAME	POSITION	MEDICAL(date)	TRAINING(type,date)

OTHER PERSONNEL (prearranged visitors, support personnel):

NAME	AGENCY/COMPANY	RESTRICTIONS

MONITORING PROCEDURES (use and employment of fixed, portable, real-time, continuous and/or periodic monitoring devices):

_____

_____

_____

_____

_____

DECONTAMINATION PROCEDURES (include sketch of exclusion, contamination reduction and support zones): PERSONNEL _____

PERSONNEL PROTECTIVE EQUIPMENT _____

SAMPLING EQUIPMENT _____

SUPPORT EQUIPMENT _____

DECON MATERIALS REQUIRED (containers, decon solutions): _____

SPECIAL HAZARDS: _____

EMERGENCY PROCEDURES

HIGH HAZARD MATERIALS (known or anticipated):

NAME	ACUTE EXPOSURE SYMPTOMS	FIRST AID

LOCATION OF NEAREST WORKING PHONE _____

OTHER EMERGENCY COMMUNICATIONS _____

EMERGENCY PHONE NUMBERS:

	NAME/LOCATION	PHONE #	PREPLAN/STANBY
AMBULANCE			
FIRE			
POLICE			
HOSPITAL			
EOD			
ENVIRONMENTAL			
UTILITIES			

ADDITIONAL RESOURCES:

NAME AGENCY/COMPANY PHONE #

ROUTE TO HOSPITAL

ALTERNATIVE

EQUIPMENT CHECKLIST

PROTECTIVE AND SAFETY EQUIPMENT (model, type, material, amount required):

SCBA SPARE CYLINDERS

ESCAPE MASK CHEM-MECH RESPIRATORS

FULL FACE HALF FACE

CANISTER CARTRIDGE

HARDHAT FACE SHIELD CHEM GOGGLES

SAFETY GLASSES EAR PROTECTION

GLOVES: SURGICAL CHEM OUTER

CHEM RESIST COVERALLS DISPOSAL COVERALLS

SPLASH APRONS SPLASH SUITS

BOOTS BOOT/SHOE COVERS

FULLY ENCAPSULATED SUITS

DOSIMETERS

FIRST AID EQUIPMENT

EYE WASH STATION FIRE EXTINGUISHER

DECON MATERIALS

SAFETY HARNESS SPECIAL TOOLS

OTHER

AFTER ACTION REPORT TO DATE

POST SITE MEDICALS

PLAN APPROVED BY DATE

APPENDIX D

SITE ENTRY - SURVEY AND RECONNAISSANCE

SITE ENTRY - SURVEY AND RECONNAISSANCE

I. INTRODUCTION

The team(s) initially entering the site is to accomplish one or more of the following objectives:

- Characterize the hazards that exist or potentially exist affecting the public health, the environment, and response personnel.
- Verify existing information and/or obtain data about the incident.
- Evaluate the need for prompt mitigation actions.
- Collect supplemental information to determine the safety requirements for personnel initially and subsequently entering the site.

Before the team enters the site, as much information as possible should be collected, depending on the time available, concerning the type(s) of hazards, degree of hazard(s), and risks which may exist. Based upon available information (shipping manifests, transportation placards, existing records, container labels, etc.) or off-site studies, the team assesses the hazards, determines the need to go on-site, and identifies initial safety requirements.

II. PRELIMINARY ON-SITE EVALUATION

The initial on-site survey is to determine, on a preliminary basis, hazardous or potentially hazardous conditions. The main effort is to rapidly identify the immediate hazards that may affect the public, response personnel, and the environment. Of major concern are the real or potential dangers - for example, fire, explosion, oxygen-deficient atmospheres, radiation, airborne contaminants, containerized or pooled hazardous substances, that could affect workers during subsequent operations.

A. Organic Vapors and Gases

If the type(s) of organic substance(s) involved in an incident is known and the material is volatile or can become airborne, air measurements for organics should be made with one or more appropriate, properly calibrated survey instruments.

When the presence or types of organic vapors/gases are unknown, instruments such as a photoionizer (HNU Systems*) and/or a portable gas chromatograph (Century Systems OVA*), operated in the total readout

*The use of any trade names does not imply their endorsement by the U.S. Environmental Protection Agency.

mode, should be used to detect organic vapors. Until specific constituents can be identified, the readout indicates total airborne subtances to which the instrument is responding. Identification of the individual vapor/gas constituents permits the instruments to be calibrated and used for more specific analysis.

Sufficient data should be obtained during the initial entry to map or screen the site for various levels of organic vapors. These gross measurements can be used on a preliminary basis to: 1) determine levels of personnel protection, 2) establish site work zones, and 3) select candidate areas for more thorough qualitative and quantitative studies.

Higher than background readings on the HNU or OVA may also indicate the displacement of oxygen or the presence of combustible vapors.

B. Inorganic Vapors and Gases

The ability to detect and quantify nonspecific inorganic vapors and gases is extremely limited. Presently, the HNU photoionizer has limited detection capability while the Century Systems has none. (See Appendix I for characteristics). If specific inorganics are known or suspected to be present, measurements should be made with appropriate instruments, if available. Colorimetric tubes can be used if substances present are known (or can be narrowed to a few) and appropriate tubes are available.

C. Radiation

Although radiation monitoring is not necessary for all responses, it should be incorporated in the initial survey where radioactive materials may be present - for example, fires at warehouses or hazardous material storage facilities, transportation incidents involving unknown materials, or abandoned waste sites.

Normal gamma radiation background is approximately 0.01 to 0.02 milliroentgen per hour (mR/hr) on a gamma survey instrument. Work can continue with elevated radiation exposure rates; however, if the exposure rate increases to 3-5 times above gamma background, a qualified health physicist should be consulted. At no time should work continue with an exposure rate of 10 mR/hr or above without the advice of a health physicist. EPA's Office of Air, Noise, and Radiation has radiation specialists in each Region, as well as at Headquarters, Montgomery, Alabama, and Las Vegas, Nevada, to assist.

The absence of gamma readings above background should not be interpreted as the complete absence of radioactivity. Radioactive materials emitting low-energy gamma, alpha, or beta radiation may be present, but for a number of reasons may not cause a response on the instrument. Unless airborne, these radioactive materials should present minimal hazard, but more thorough surveys should be conducted

as site operations continue to completely eliminate the presence of any radioactive material.

D. Oxygen Deficiency

At sea level, ambient air must contain at least 19.5% by volume of oxygen. At lower precentages, air-supplied respiratory protective equipment is needed. Oxygen measurements are of particular importance for work in enclosed spaces, low-lying areas, or in the vicinity of accidents that have produced heavier-than-air vapors, which could displace ambient air. These oxygen-deficient areas are also prime locations for taking further organic vapor and combustible gas measurements, since the air has been displaced by other substances. Oxygen-enriched atmospheres increase the potential for fires.

E. Combustible Gases

The presence or absence of combustible vapors or gases must be determined. If readings approach or exceed 10% of the lower explosive limit (LEL), extreme caution should be exercised in continuing the investigation. If readings approach or exceed 25% LEL, personnel should be withdrawn immediately. Before resuming any on-site activities, project personnel in consultation with experts in fire or explosion prevention must develop procedures for continuing operations.

F. Visual Observations

While on-site, the initial entry team should make visual observations which would help in evaluating site hazards - for example, dead fish or other animals; land features; wind direction; labels on containers indicating explosive, flammable, toxic, or corrosive materials; conditions conducive to splash or contact with unconfined liquids, sludges, or solids; and other general conditions.

G. Direct-Reading Instruments

A variety of toxic air pollutants, (including organic and inorganic vapors, gases, or particulates) can be produced at, for example, abandoned waste sites; fires at chemical manufacturing, storage, reprocessing, or formulating facilities; or fires involving pesticides. Direct-reading field instruments will not detect or measure all of these substances. Thus, negative readings should not be interpretec as the complete absence of airborne toxic substances. Verification of negative results can only be done by collecting air samples and analyzing them in a laboratory.

III. OTHER CONSIDERATIONS

A. Initial Surveys

In general, the initial entry is considered a relatively rapid screening process for collecting preliminary data on site hazards. The time needed to conduct the initial survey depends on the urgency of the situation, type of incident, information needed, size of site, availability of resources, level of protection required for initial entry personnel, etc. Consequently, initial surveys may need hours or days to complete and consist of more than one entry.

B. Priority for Initial Entry Monitoring

Of immediate concern to initial entry personnel are atmospheric conditions which could affect their immediate safety. These conditions are airborne toxic substances, combustible gases or vapors, lack of oxygen, and to a lesser extent, ionizing radiation. Priorities for monitoring these potential hazards should be established after a careful evaluation of conditions.

When the type(s) of material(s) involved in an incident is identified and its release into the environment suspected or known, the material's chemical/physical properties and the prevailing weather conditions may help determine the order of monitoring. An unknown substance(s) or situation(s) presents a more difficult monitoring problem.

In general, for poorly ventilated spaces - buildings, ship's holds, boxcars, or bulk tanks - which must be entered, combustible vapors/gases and oxygen-deficient atmospheres should be monitored first with team members wearing, as a minimum, Level B protective equipment (Levels of Protection are described in Part 5). Toxic gases/vapors and radiation, unless known not to be present, should be measured as the next priority.

For open, well-ventilated areas, combustible gases and oxygen deficiency are lesser hazards, and require lower priority. However, areas of lower elevation on-site (such as ditches and gulleys) and downwind areas may have combustible gas mixtures, in addition to toxic vapors or gases, and lack sufficient oxygen to sustain life. Entry teams should approach and monitor whenever possible from the upwind area.

C. Periodic Monitoring

The monitoring surveys made during the initial site entry phase are for a preliminary evaluation of atmospheric hazards. In some situations, the information obtained may be sufficient to preclude additional monitoring - for example, a chlorine tank determined to be releasing no chlorine. Materials detected during the initial site

survey call for a more comprehensive evaluation of hazards and analyses for specific components. A program must be established for monitoring, sampling, and evaluating hazards for the duration of site operations. Since site activities and weather conditions change, a continuous program to monitor atmospheric changes must be implemented utilizing a combination of stationary sampling equipment, personnel monitoring devices, and periodic area monitoring with direct-reading instruments.

D. Peripheral Monitoring

Whenever possible, atmospheric hazards in the areas adjacent to the on-site zone should be monitored with direct-reading instruments, and air samples should be taken before the initial entry for on-site monitoring. Negative instrument readings off-site should not be construed as definite indications of on-site conditions, but only another piece of information to assist in the preliminary evluation.

E. Monitoring Instruments

It is imperative that personnel using monitoring instruments be thoroughly familiar with their use, limitations, and operating characteristics. All instruments have inherent constraints in their ability to detect and/or quantify the hazards for which they were designed. Unless trained personnel use instruments and assess data readout, air hazards can be grossly misinterpreted, endangering the health and safety of response personnel. In addition, only intrinsically safe instruments should be used, until the absence of combustible gases or vapors can be confirmed.

F. Ambient Atmospheric Concentrations

Any indication of atmospheric hazards - toxic substances, combustible gases, lack of oxygen, radiation, and other specific materials - should be viewed as a sign to proceed with care and deliberation. Readings indicating nonexplosive atmospheres, low concentrations of toxic substances, or other conditions may increase or decrease suddenly, changing the associated risks. Extreme caution should be exercised in continuing surveys when atmospheric hazards are indicated.

TABLE 4-1

ATMOSPHERIC HAZARD GUIDELINES

Monitoring Equipment	Hazard	Ambient Level	Action
Combustible gas indicator	Explosive atmosphere	< 10% LEL	Continue investigation.
		10%-25%	Continue on-site monitoring with extreme caution as higher levels are encountered.
		> 25% LEL	Explosion hazard; withdraw from area immediately.
Oxygen concentration meter	Oxygen	< 19.5%	Monitor wearing SCBA. NOTE: Combustible gas readings are not valid in atmospheres with < 19.5% oxygen.
		19.5%-25%	Continue investigation with caution. SCBA not needed, based on oxygen content only.
		> 25.0%	Discontinue inspection; fire hazard potential. Consult specialist.
Radiation survey	Radiation	< 1 mR/hr	Continue investigation. If radiation is detected above background levels, this signifies the presence of possible radiation sources; at this level, more thorough monitoring is advisable. Consult with a health physicist.
		> 10 mR/hr	Potential radiation hazard; evacuate site. Continue monitoring only upon the advice of a health physicist.
Colorimetric tubes	Organic and inorganic vapors/gases	Depends on species	Consult standard reference manuals for air concentrations/ toxicity data.

TABLE 4-1 (Continued)

HNU photoionizer	Organic vapors/gases	1) Depends on species	Consult standard reference manuals for air concentrations/ toxicity data.
		2) Total response mode	Consult EPA Standard Operating Procedures.
Organic vapor analyzer	Organic	1) Depends on species	Consult standard reference manuals for air concentrations/ toxicity data.
		2) Total response mode	Consult EPA Standard Operating Procedures.

APPENDIX E

RATIONALE FOR RELATING TOTAL ATMOSPHERIC VAPOR/GAS CONCENTRATIONS TO THE SELECTION OF THE LEVEL OF PROTECTION

RATIONALE FOR RELATING TOTAL ATMOSPHERIC VAPOR/GAS CONCENTRATIONS TO THE SELECTION OF THE LEVEL OF PROTECTION

I. INTRODUCTION

The objective of using total atmospheric vapor/gas concentrations for determining the appropriate Level of Protection is to provide a numerical criterion for selecting Level A, B, or C. In situations where the presence of vapors or gases is not known, or if present, the individual components are unknown, personnel required to enter that environment must be protected. Until the constituent substances and corresponding atmospheric concentrations of vapor, gas, or particulate can be determined and respiratory and/or body protection related to the toxicological properties of the identified substances, total vapor/gas concentration, with judicious interpretation, can be used as a guide for selecting personnel protection equipment.

Although total vapor/gas concentration measurements are useful to a qualified professional for the selection of protection equipment, caution should be exercised in interpretation. An instrument does not respond with the same sensitivity to several vapor/gas contaminants as it does to a single contaminant. Also since total vapor/gas field instruments see all contaminants in relation to a specific calibration gas, the concentration of unknown gases or vapors may be over - or under-estimated.

Suspected carcinogens, particulates, highly hazardous substances, or other substances that do not elicit an instrument response may be known or believed to be present. Therefore, the protection level should not be based solely on the total vapor/gas criterion. Rather, the level should be selected case-by-case, with special emphasis on potential exposure and chemical and toxicological characteristics of the known or suspected material.

II. FACTORS FOR CONSIDERATION

In utilizing total atmospheric vapor/gas concentrations as a guide for selecting a Level of Protection, a number of other factors should also be considered:

- The uses, limitations, and operating characteristics of the monitoring instruments must be recognized and understood. Instruments such as the HNU Photoionizer, Century Organic Vapor Analyzer (OVA), MIRAN Infrared Spectrophotometer, and others do not respond identically to the same concentration of a substance or respond to all substances. Therefore, experience, knowledge, and good judgment must be used to complement the data obtained with instruments.

- Other hazards may exist such as gases not detected by the HNU or OVA, (i.e. phosgene, cyanides, arsenic, chlorine), explosives, flammable materials, oxygen deficiency, liquid/solid particles, and liquid or solid chemicals.

- Vapors/gases with very low toxicities could be present.

- The risk to personnel entering an area must be weighed against the need for entering. Although this assessment is largely a value judgment, it requires a conscientious balancing of the variables involved and the risk to personnel against the need to enter an unknown environment.

- The knowledge that suspected carcinogens or substances extremely toxic or destructive to skin are present or suspected to be present (which may not be reflected in total vapor/gas concentration) requires an evaluation of factors such as the potential for exposure, chemical characteristics of the material, limitation of instruments, and other considerations specific to the incident.

- What needs to be done on-site must be evaluated. Based upon total atmospheric vapor concentrations, Level C protection may be judged adequate; however, tasks such as moving drums, opening containers, and bulking of materials, which increase the probability of liquid splashes or generation of vapors, gases, or particulates, may require a higher level of protection.

- Before any respiratory protective apparatus is issued, a respiratory protection program must be developed and implemented according to recognized standards (ANSI Z88.2-1980).

III. LEVEL A PROTECTION (500 to 1,000 PPM ABOVE BACKGROUND)

Level A protection provides the highest degree of respiratory tract, skin, and eye protection if the inherent limitations of the personnel protective equipment are not exceeded. The range of 500 to 1,000 parts per million (ppm) total vapors/gases concentration in air was selected based on the following criteria:

- Although Level A provides protection against air concentrations greater than 1,000 ppm for most substances, an operational restriction of 1,000 ppm is established as a warning flag to:

  -- evaluate the need to enter environments with unknown concentrations greater than 1,000 ppm

  -- identify the specific constituents contributing to the total concentration and their associated toxic properties

  -- determine more precisely concentrations of constituents

-- evaluate the calibration and/or sensitivity error associated with the instrument(s)

-- evaluate instrument sensitivity to wind velocity, humidity temperature, etc.

- A lower limit of 500 ppm total vapors/gases in air was selected as the value to consider upgrading from Level B to Level A. This concentration was selected to fully protect the skin until the constituents can be identified and measured and substances affecting the skin excluded.

- The range of 500 to 1,000 ppm is sufficiently conservative to provide a safe margin of protection if readings are low due to instrument error, calibration, and sensitivity; if higher than anticipated concentrations occur; and if substances highly toxic to the skin are present.

With properly operating portable field equipment, ambient air concentrations approaching 500 ppm have not routinely been encountered on hazardous waste sites. High concentrations have been encountered only in closed buildings, when containers were being opened, when personnel were working in the spilled contaminants, or when organic vapors/gases were released in transportation accidents. A decision to require Level A protection should also consider the negative aspects: higher probability of accidents due to cumbersome equipment, and most importantly, the physical stress caused by heat buildup in fully encapsulating suits.

LEVEL B PROTECTION (5 to 500 ABOVE BACKGROUND)

Level B protection is the minimum Level of Protection recommended for initially entering an open site where the type(s), concentration(s), and presence of airborne vapors are unknown. This Level of Protection provides a high degree of respiratory protection. Skin and eyes are also protected, although a small portion of the body (neck and sides of head) may be exposed. The use of a separate hood or hooded, chemical-resistant jacket would further reduce the potential for exposure to this area of the body. Level B impermeable protective clothing also increases the probability of heat stress.

A limit of 500 ppm total atmospheric vapor/gas concentration on portable field instruments has been selected as the upper restriction on the use of Level B. Although Level B personnel protection should be adequate for most commonly encountered substances at air concentrations higher than 500 ppm, this limit has been selected as a decision point for a careful evaluation of the risks associated with higher concentrations. These factors should be considered:

- The necessity for entering unknown concentrations higher than 500 ppm wearing Level B protection.

- The probability that substance(s) present are severe skin hazards.

- The work to be done and the increased probability of exposure.
- The need for qualitative and quantitative identification of the specific components.
- Inherent limitations of the instruments used for air monitoring.
- Instrument sensitivity to winds, humidity, temperature, and other factors.

V. LEVEL C PROTECTION (BACKGROUND TO 5 PPM ABOVE BACKGROUND)

Level C provides skin protection identical to Level B, assuming the same type of chemical protective clothing is worn, but lesser protection against inhalation hazards. A range of background to 5 ppm above ambient background concentrations of vapors/gases in the atmosphere has been established as guidance for selecting Level C protection. Concentrations in the air of unidentified vapors/gases approaching or exceeding 5 ppm would warrant upgrading respiratory protection to a self-contained breathing apparatus.

A full-face, air-purifying mask equipped with an organic vapor canister (or a combined organic vapor/particulate canister) provides protection against low concentrations of most common organic vapors/gases. There are some substances against which full-face, canister-equipped masks do not protect, or substances that have very low Threshold Limit Values or Immediately Dangerous to Life or Health concentrations. Many of the latter substances are gases or liquids in their normal state. Gases would only be found in gas cylinders, while the liquids would not ordinarily be found in standard containers or drums.

Every effort should be made to identify the individual constituents (and the presence of particulates) contributing to the total vapor readings of a few parts per million. Respiratory protective equipment can then be selected accordingly. It is exceedingly difficult, however, to provide constant, real-time identification of all components in a vapor cloud with concentrations of a few parts per million at a site where ambient concentrations are constantly changing. If highly toxic substances have been ruled out, but ambient levels of a few parts per million persist, it is unreasonable to assume only self-contained breathing apparatus should be worn. The continuous use of air-purifying masks in vapor/gas concentrations of a few parts per million gives a reasonable assurance that the respiratory tract is protected, provided that the absence of highly toxic substances has been confirmed.

Full-face, air-purifying devices provide respiratory protection against most vapors at greater than 5 ppm; however, until more definitive qualitative information is available, concentration(s) greater than 5 ppm indicates that a higher level of respiratory protection should be used. Also, unanticipated transient excursions may increase the concentrations in

the environment above the limits of air-purifying devices. The increased probability of exposure due to the work being done may require Level B protection, even though ambient levels are low.

VI. INSTRUMENT SENSITIVITY

Although the measurement of total vapor/gas concentrations can be a useful adjunct to professional judgment in the selection of an appropriate Level of Protection, caution should be used in the interpretation of the measuring instrument's readout. The response of an instrument to a gas or vapor cloud containing two or more substances does not provide the same sensitivity as measurements involving the individual pure constituents. Hence the instrument readout may overestimate or underestimate the concentration of an unknown composite cloud. This same type of inaccuracy could also occur in measuring a single unknown substance with the instrument calibrated to a different substance. The idiosyncrasies of each instrument must be considered in conjunction with the other parameters in selecting the protection equipment needed.

Using the total vapor/gas concentration as a criterion used to determine Levels of Protection should provide protection against concentrations greater than the instrument's readout. However, when the upper limits of Level C and B are approached, serious consideration should be given to selecting a higher Level of Protection. Cloud constituent(s) must be identified as rapidly as possible and Levels of Protection based on the toxic properties of the specific substance(s) identified.

APPENDIX F

SITE CONTROL - DECONTAMINATION

SITE CONTROL - DECONTAMINATION

I. INTRODUCTION

Personnel responding to hazardous substance incidents may become contaminated in a number of ways, including:

- Contacting vapors, gases, mists, or particulates in the air.
- Being splashed by materials while sampling or opening containers.
- Walking through puddles of liquids or on contaminated soil.
- Using contaminated instruments or equipment.

Protective clothing and respirators help prevent the wearer from becoming contaminated or inhaling contaminants, while good work practices help reduce contamination on protective clothing, instruments, and equipment.

Even with these safeguards, contamination may occur. Harmful materials can be transferred into clean areas, exposing unprotected personnel. Or in removing contaminated clothing, personnel may contact contaminants on the clothing and/or inhale them. To prevent such occurrences, methods to reduce contamination and decontamination procedures must be developed and implemented before anyone enters a site and must continue (modified when necessary) throughout site operations.

Decontamination consists of physically removing contaminants and/or changing their chemical nature to innocuous substances. How extensive decontamination must be depends on a number of factors, the most important being the type of contaminants involved. The more harmful the contaminant the more extensive and thorough decontamination must be. Less harmful contaminants may require less decontamination. Combining decontamination, the correct method of doffing personnel protective equipment, and the use of site work zones minimizes cross-contamination from protective clothing to wearer, equipment to personnel, and one area to another. Only general guidance can be given on methods and techniques for decontamination. The exact procedure to use must be determined after evaluating a number of factors specific to the incident.

II. PRELIMINARY CONCERNS

A. Initial Planning

The initial decontamination plan assumes all personnel and equipment leaving the Exclusion Zone (area of potential contamination) are grossly contaminated. A system is then set up to wash and rinse,

at least once, all the personnel protective equipment worn. This is done in combination with a sequential doffing of equipment, starting at the first station with the most heavily contaminated item and progressing to the last station with the least contaminated article. Each piece of clothing or operation requires a separate station.

The spread of contaminants during the washing/doffing process is further reduced by separating each decontamination station by a minimum of 3 feet. Ideally, contamination should decrease as a person moves from one station to another farther along in the line.

While planning site operations, methods should be developed to prevent the contamination of people and equipment. For example, using remote sampling techniques, not opening containers by hand, bagging monitoring instruments, using drum grapplers, watering down dusty areas, and not walking through areas of obvious contamination would reduce the probability of becoming contaminated and require a less elaborate decontamination procedure.

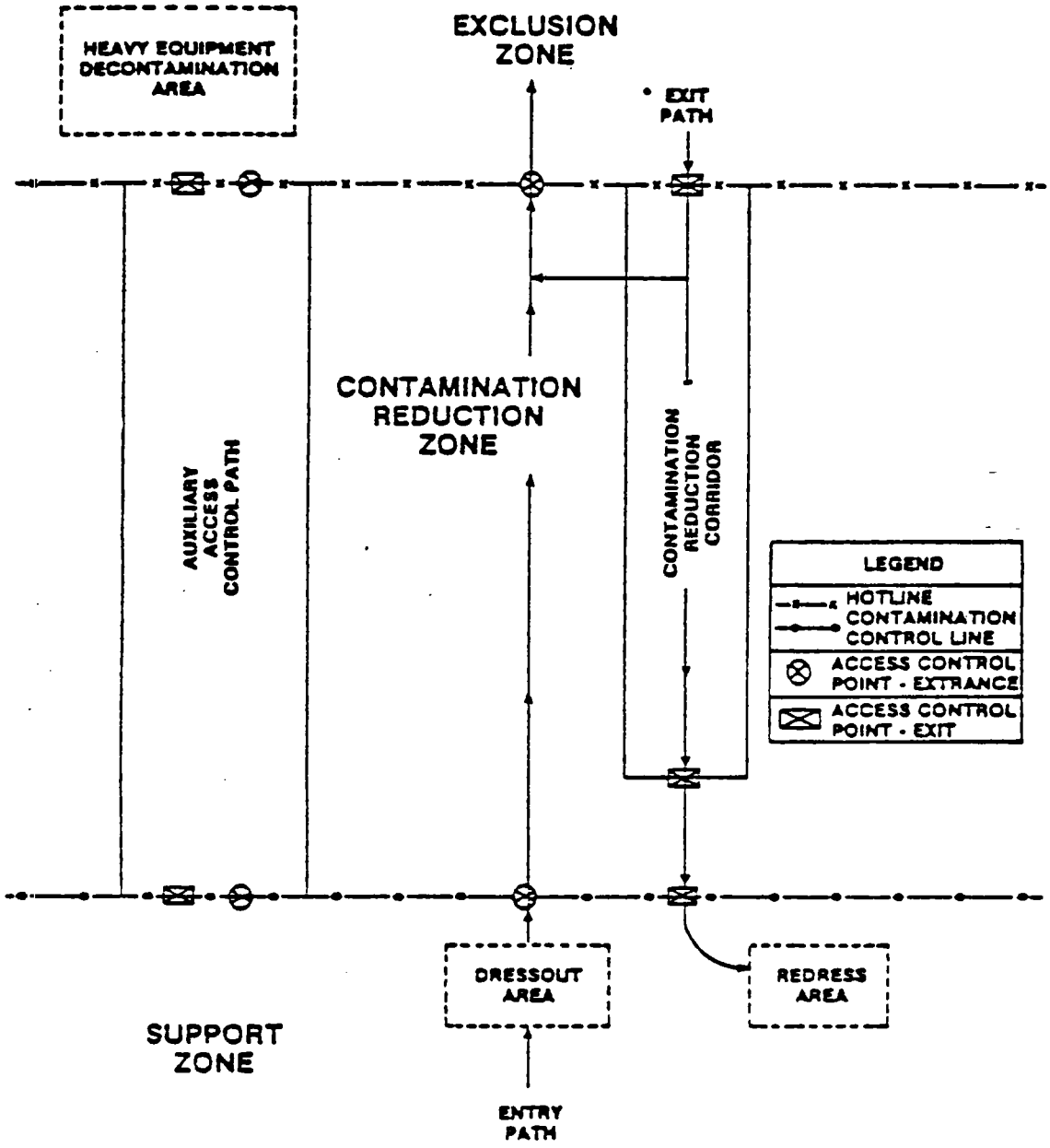
The initial decontamination plan is based on a worst-case situation (if no information is available about the incident). Specific conditions at the site are then evaluated, including:

- Type of contaminant.
- The amount of contamination.
- Levels of protection required.
- Type of protective clothing worn.

The initial system is modified, eliminating unnecessary stations or otherwise adapting it to site conditions. For instance, the initial plan might require a complete wash and rinse of chemical protective garments. If disposable garments are worn, the wash/rinse step could be omitted. Wearing disposable boot covers and gloves could eliminate washing and rinsing both gloves and disposable boots and reduce the number of stations needed.

B. Contamination Reduction Corridor

An area within the Contamination Reduction Zone is designated the Contamination Reduction Corridor (CRC). The CRC controls access into and out of the Exclusion Zone and confines personnel decontamination activities to a limited area. The size of the corridor depends on the number of stations in the decontamination procedure, overall dimensions of work control zones, and amount of space available at the site. A corridor of 75 feet by 15 feet should be adequate for full decontamination. Whenever possible, it should be a straight path.



CONTAMINATION REDUCTION ZONE LAYOUT
FIGURE 7-1

The CRC boundaries should be conspicuously marked, with entry and exit restricted. The far end is the hotline - the boundary between the Exclusion Zone and the Contamination Reduction Zone. Personnel exiting the Exclusion Zone must go through the CRC. Anyone in the CRC should be wearing the Level of Protection designated for the decontamination crew. Another corridor may be required for the entrance and exit of heavy equipment needing decontamination. Within the CRC, distinct areas are set aside for decontamination of personnel, portable field equipment, removed clothing, etc. These areas should be marked and personnel restricted to those wearing the appropriate Level of Protection. All activities within the corridor are confined to decontamination.

Personnel protective clothing, respirators, monitoring equipment, sampling supplies, etc. are all maintained outside of the CRC. Personnel don their protective equipment away from the CRC and enter the Exclusion Zone through a separate access control point at the hotline.

III. EXTENT OF DECONTAMINATION REQUIRED

A. Modifications of Initial Plan

The original decontamination plan must be adapted to specific conditions found at incidents. These conditions may require more or less personnel decontamination than planned, depending on a number of factors.

1. Type of Contaminant

The extent of personnel decontamination depends on the effects the contaminants have on the body. Contaminants do not exhibit the same degree of toxicity (or other hazard). The more toxic a substance is the more extensive or thorough decontamination must be. Whenever it is known or suspected that personnel can become contaminated with highly toxic or skin-destructive substances, a full decontamination procedure should be followed. If less hazardous materials are involved, the procedure can be downgraded.

2. Amount of Contamination

The amount of contamination on protective clothing is usually determined visually. If it is badly contaminated, a thorough decontamination is generally required. Gross material remaining on the protective clothing for any extended period of time may degrade or permeate it. This likelihood increases with higher air concentrations and greater amounts of liquid contamination. Gross contamination also increases the probability of personnel contact. Swipe tests may help determine the type and quantity of surface contaminants.

3. Level of Protection

The Level of Protection and specific pieces of clothing worn determine on a preliminary basis the layout of the decontamination line. Each Level of Protection incorporates different problems in decontamination and doffing of the equipment. For example: decontamination of the harness straps and backpack assembly of the self-contained breathing apparatus is difficult. A butyl rubber apron worn over the harness makes decontamination easier. Clothing variations and different Levels of Protection may require adding or deleting stations in the original decontamination procedure.

4. Work Function

The work each person does determines the potential for contact with hazardous materials. In turn, this dictates the layout of the decontamination line. Observers, photographers, operators of air samplers, or others in the Exclusion Zone performing tasks that will not bring them in contact with contaminants may not need, for example, to have their garments washed and rinsed. Others in the Exclusion Zone with a potential for direct contact with the hazardous material will require more thorough decontamination. Different decontamination lines could be set up for different job functions, or certain stations in a line could be omitted for personnel performing certain tasks.

5. Location of Contamination

Contamination on the upper areas of protective clothing poses a greater risk to the worker because volatile compounds may generate a hazardous breathing concentration both for the worker and for the decontamination personnel. There is also an increased probability of contact with skin when doffing the upper part of clothing.

6. Reason for Leaving Site

The reason for leaving the Exclusion Zone also determines the need and extent of decontamination. A worker leaving the Exclusion Zone to pick up or drop off tools or instruments and immediately returning may not require decontamination. A worker leaving to get a new air cylinder or change a respirator or canisters, however, may require some degree of decontamination. Individuals departing the CRC for a break, lunch, end of day, etc., must be thoroughly decontaminated.

B. Effectiveness of Decontamination

There is no method to immediately determine how effective decontamination is in removing contaminants. Discolorations, stains, corrosive effects, and substances adhering to objects may indicate contaminants have not been removed. However, observable effects only

indicate surface contamination and not permeation (absorption) into clothing. Also many contaminants are not easily observed.

A method for determining effectiveness of surface decontamination is swipe testing. Cloth or paper patches - swipes - are wiped over predetermined surfaces of the suspect object and analyzed in a laboratory. Both the inner and outer surfaces of protective clothing should be swipe tested. Positive indications of both sets of swipes would indicate surface contamination has not been removed and substances have penetrated or permeated through the garment. Swipe tests can also be done on skin or inside clothing. Permeation of protective garments requires laboratory analysis of a piece of the material. Both swipe and permeation testing provide after-the-fact information. Along with visual observations, results of these tests can help evaluate the effectiveness of decontamination.

C. Equipment

Decontamination equipment, materials, and supplies are generally selected based on availability. Other considerations are ease of equipment decontamination or disposability. Most equipment and supplies can be easily procured. For example, soft-bristle scrub brushes or long-handle brushes are used to remove contaminants. Water in buckets or garden sprayers is used for rinsing. Large galvanized wash tubs or stock tanks can hold wash and rinse solutions. Children's wading pools can also be used. Large plastic garbage cans or other similar containers lined with plastic bags store contaminated clothing and equipment. Contaminated liquids can be stored temporarily in metal or plastic cans or drums. Other gear includes paper or cloth towels for drying protective clothing and equipment.

D. Decontamination Solution

Personnel protective equipment, sampling tools, and other equipment are usually decontaminated by scrubbing with detergent-water using a soft-bristle brush followed by rinsing with copious amounts of water. While this process may not be fully effective in removing some contaminants (or in a few cases, contaminants may react with water), it is a relatively safe option compared with using a chemical decontaminating solution. This requires that the contaminant be identified. A decon chemical is then needed that will change the contaminant into a less harmful substance. Especially troublesome are unknown substances or mixtures from a variety of known or unknown substances. The appropriate decontamination solution must be selected in consultation with an experienced chemist.

E. Establishment of Procedures

Once decontamination procedures have been established, all personnel requiring decontamination must be given precise instructions (and practice, if necessary). Compliance must be frequently checked. The

time it takes for decontamination must be ascertained. Personnel wearing SCBA's must leave their work area with sufficient air to walk to CRC and go through decontamination.

IV. DECONTAMINATION DURING MEDICAL EMERGENCIES

A. Basic Considerations

Part of overall planning for incident response is managing medical emergencies. The plan should provide for:

- Some response team members fully trained in first aid and CPR.
- Arrangements with the nearest medical facility for transportation and treatment of injured, and for treatment of personnel suffering from exposure to chemicals.
- Consultation services with a toxicologist.
- Emergency eye washes, showers, and/or wash stations.
- First aid kits, blankets, stretcher, and resuscitator.

In addition, the plan should have established methods for decontaminating personnel with medical problems and injuries. Their is the possibility that the decontamination may aggravate or cause more serious health effects. If prompt life-saving first aid and/or medical treatment is required, decontamination procedures should be omitted. Whenever possible, response personnel should accompany contaminated victims to the medical facility to advise on matters involving decontamination.

B. Physical Injury

Physical injuries can range from a sprained ankle to a compound fracture, from a minor cut to massive bleeding. Depending on the seriousness of the injury, treatment may be given at the site by trained response personnel. For more serious injuries, additional assistance may be required at the site or the victim may have to be treated at a medical facility.

Life-saving care should be instituted immediately without considering decontamination. The outside garments can be removed (depending on the weather) if they do not cause delays, interfere with treatment, or aggravate the problem. Respiratory masks and backpack assemblies must always be removed. Fully encapsulating suits or chemical-resistant clothing can be cut away. If the outer contaminated garments cannot be safely removed, the individual should be wrapped in plastic, rubber, or blankets to help prevent contaminating the inside of ambulances and/or medical personnel. Outside garments are then removed at the medical

facility. No attempt should be made to wash or rinse the victim. One exception would be if it is known that the individual has been contaminated with an extremely toxic or corrosive material which could also cause severe injury or loss of life. For minor medical problems or injuries, the normal decontamination procedure should be followed.

C. Heat Stress

Heat-related illnesses range from heat fatigue to heat stroke, the most serious. Heat stroke requires prompt treatment to prevent irreversible damage or death. Protective clothing may have to be cut off. Less serious forms of heat stress require prompt attention or they may lead to a heat stroke. Unless the victim is obviously contaminated, decontamination should be omitted or minimized and treatment begun immediately.

D. Chemical Exposure

Exposure to chemicals can be divided into two categories:

- Injuries from direct contact, such as acid burns or inhalation of toxic chemicals.

- Potential injury due to gross contamination on clothing or equipment.

For the contaminant inhaled, treatment can only be by qualified physicians. If the contaminant is on the skin or in the eyes, immediate measures must be taken to counteract the substance's effect. First aid treatment usually is flooding the affected area with water; however, for a few chemicals, water may cause more severe problems.

When protective clothing is grossly contaminated, contaminants may be transferred to treatment personnel or the wearer and cause injuries. Unless severe medical problems have occurred simultaneously with splashes, the protective clothing should be washed off as rapidly as possible and carefully removed.

V. PROTECTION FOR DECONTAMINATION WORKERS

The Level of Protection worn by decontamination workers is determined by:

- Expected or visible contamination on workers.

- Type of contaminant and associated respiratory and skin hazards.

- Total vapor/gas concentrations in the CRC.

- Particulates and specific inorganic or organic vapors in the CRC.

- Results of swipe tests.

- The presence (or suspected presence) of highly toxic or skin-destructive materials.

A. Level C Use

Level C includes a full-face, canister-type air-purifying respirator, hard hat with face shield (if splash is a problem), chemical-resistant boots and gloves, and protective clothing. The body covering recommended is chemical-resistant overalls with an apron, or chemical-resistant overalls and jacket.

A face shield is recommended to protect against splashes because respirators alone may not provide this protection. The respirator should have a canister approved for filtering any specific known contaminants such as ammonia, organic vapors, acid gases, and particulates.

B. Level B Use

In situations where site workers may be contaminated with unknowns, highly volatile liquids, or highly toxic materials, decontamination workers should wear Level B protection.

Level B protection includes SCBA, hard hat with face shield, chemical-resistant gloves, and protective covering. The clothing suggested is chemical-resistant overalls, jacket, and a rubber apron. The rubber apron protects the SCBA harness assembly and regulator from becoming contaminated.

VI. DECONTAMINATION OF EQUIPMENT

Insofar as possible, measures should be taken to prevent contamination of sampling and monitoring equipment. Sampling devices become contaminated, but monitoring instruments, unless they are splashed, usually do not. Once contaminated, instruments are difficult to clean without damaging them. Any delicate instrument which cannot be decontaminated easily should be protected while it is being used. It should be bagged, and the bag taped and secured around the instrument. Openings are made in the bag for sample intake.

A. Decontamination Procedures

1. Sampling devices

Sampling devices require special cleaning. The EPA Regional Laboratories can provide information on proper decontamination methods.

2. Tools

Wooden tools are difficult to decontaminate because they absorb chemicals. They should be kept on site and handled only by protected workers. At the end of the response, wooden tools should be discarded. For decontaminating other tools, Regional Laboratories should be consulted.

3. Respirators

Certain parts of contaminated respirators, such as the harness assembly and leather or cloth components, are difficult to decontaminate. If grossly contaminated, they may have to be discarded. Rubber components can be soaked in soap and water and scrubbed with a brush. Regulators must be maintained according to manufacturer's recommendations. Persons responsible for decontaminating respirators should be thoroughly trained in respirator maintenance.

4. Heavy Equipment

Bulldozers, trucks, back-hoes, bulking chambers, and other heavy equipment are difficult to decontaminate. The method generally used is to wash them with water under high pressure and/or to scrub accessible parts with detergent/water solution under pressure, if possible. In some cases, shovels, scoops, and lifts have been sand blasted or steam cleaned. Particular care must be given to those components in direct contact with contaminants such as tires and scoops. Swipe tests should be utilized to measure effectiveness.

B. Sanitizing of Personnel Protective Equipment

Respirators, reusable protective clothing, and other personal articles not only must be decontaminated before being reused, but also sanitized. The inside of masks and clothing becomes soiled due to exhalation, body oils, and perspiration. The manufacturer's instructions should be used to sanitize the respirator mask. If practical, protective clothing should be machine washed after a thorough decontamination; otherwise it must be cleaned by hand.

C. Persistent Contamination

In some instances, clothing and equipment will become contaminanted with substances that cannot be removed by normal decontamination procedures. A solvent may be used to remove such contamination from equipment if it does not destroy or degrade the protective material. If persistent contamination is expected, disposable garments should be used. Testing for persistent contamination of protective clothing and appropriate decontamiantion must be done by qualified laboratory personnel.

D. Disposal of Contaminated Materials

All materials and equipment used for decontamination must be disposed of properly. Clothing, tools, buckets, brushes, and all other equipment that is contaminated must be secured in drums or other containers and labeled. Clothing not completely decontaminated on-site should be secured in plastic bags before being removed from the site.

Contaminated wash and rinse solutions should be contained by using step-in-containers (for example, child's wading pool) to hold spent solutions. Another containment method is to dig a trench about 4 inches deep and line it with plastic. In both cases the spent solutions are transferred to drums, which are labeled and disposed of with other substances on site.

VII. ANNEXES

Annex 1, 2, and 3 describe basic decontamination procedures for a worker wearing Level A, B, or C protection. The basic decontamination lines (Situation 1), consisting of approximately 19 stations, are almost identical except for changes necessitated by different protective clothing or respirators. For each annex, three specific situations are described in which the basic (or full decontamination) procedure is changed to take into account differences in the extent of contamination, the accompanying changes in equipment worn, and other factors. The situations illustrate decontamination setups based on known or assumed conditions at an incident. Many other variations are possible.

Annex 4 describes a minimum layout for personnel decontamination. The number of individual stations have been reduced. Although the decontamination equipment and amount of space required is less than needed in the procedures previously described, there is also a much higher probability of cross-contamination.

EQUIPMENT DECONTAMINATION

I. GENERAL

Although contamination avoidance is the best posture to adopt at a hazardous material site, certain equipment used in remedial actions or sampling will unavoidably become contaminated. These items must either be properly decontaminated before being removed from the site or in the case in sampling equipment thoroughly cleaned before the next use. Wherever possible, disposable sampling equipment should be utilized to minimize the quantities of equipment to be cleaned and volume of decontaminants and rinse solutions to be disposed of. Likewise, disposable plastic tarpaulins can be placed over certain items of equipment to minimize subsequent cleaning.

The decontamination of vehicles and large pieces of equipment, such as pumps, must be done on a wash pad constructed so that cleaning solutions and wash water can be recycled or collected for later disposal. A thorough inspection of equipment, supplemented by a swipe test is appropriate, should be the governing factor for length and method of decontamination. It is important that all portions of the equipment including the under carriage, chassis, and cab be thoroughly cleaned. Air filters on equipment utilized in or around the exclusionary zone should be considered highly contaminated and removed and replaced prior to leaving the site. Porous items such as wooden truck beds, cloth hoses, and wooden handles cannot, in many instances, be properly cleaned.

Steam cleaning or high pressure spraying utilizing water with a general purpose low sudsing soap or detergent, to improve wetting effects, is the decontamination method of choice. Physical scrubbing by disposable or easily decontaminated brushes may be necessary to loosen materials. In most instances hot water is more effective than cold. Flushing should be done under high pressure, taking care not to damage items on the equipment such as dials and gauges and loosely hanging wires or hoses.

II. DECONTAMINANTS

As stated above, steam or hot water with detergent is the decontaminant of choice. However, in some cases, it may be necessary to utilize a special solution or combination of solutions to affect a thorough decontamination. It is important that whatever decontaminant is utilized, its possible reactivity and suitability for the hazardous materials involved be carefully evaluated. Other general decontaminants that might be utilized are categorized in the accompanying table. Additional specialized decontaminants that may be considered include: ethanol, acetone and solvents such as 1, 1, 1-trichloroethane for small items used in sampling; supertropical bleach (STB); DS2, a mixture of diethylenetriamine (70%), ethylene glycol monmethyl ether (28%), and sodium hydroxide (2%); sodium hydroxide (caustic soda); chelating agents such as ETDA, citric acid, tartaric acid and oxalic acid. Biological contaminants have been decontaminated utilizing: betapropiolactone (BPL); formaldehyde solution; ethylene oxide-fluorinated hydrocarbon mixture; peracetic acid; and strong bleaches and caustics. These decontaminants all require special care in their handling and use.

DECON SOLUTION	REMARKS
1. Sodium Carbonate (Washing Soda)	5% - 10% aqueous solution, good water softening agent, effective for inorganic acids
2. Sodium Bicarbonate (Baking Soda)	5% - 10% aqueous solution, amphoteric - effective with most acids and bases
3. Trisodium Phosphate (TSP, Oakite)	5% aqueous solution, good water softening agent - detergent, general rinse solution
4. Combination	An aqueous solution of 5% Sodium Carbonate and 5% Trisodium Phosphate
5. Calcium Hypochlorite (HTH)	10% aqueous solution, disinfectant, bleaching and oxidizing agent care required in storage, mixing and application

ANNEX 1

LEVEL A DECONTAMINATION

A. EQUIPMENT WORN

The full decontamination procedure outlined is for workers wearing Level A protection (with taped joints between gloves, boots, and suit) consisting of:

- Fully encapsulating suit with integral boots and gloves.
- Self-contained breathing apparatus.
- Hard hat (optional).
- Chemical-resistant, steel toe and shank boots.
- Boot covers.
- Inner and outer gloves.

B. PROCEDURE FOR FULL DECONTAMINATION

Station 1: Segregated Equipment Drop

Deposit equipment used on-site (tools, sampling devices and containers, monitoring instruments, radios, clipboards, etc.) on plastic drop cloths or in different containers with plastic liners. Each will be contaminated to a different degree. Segregation at the drop reduces the probability of cross-contamination.

Equipment: various size containers
plastic liners
plastic drop cloths

Station 2: Boot Cover and Glove Wash

Scrub outer boot covers and gloves with decon solution or detergent/water.

Equipment: container (20-30 gallons)
decon solution
or
detergent water
2-3 long-handle, soft-bristle scrub brushes

Station 3: Boot Cover and Glove Rinse

Rinse off decon solution from Station 2 using copious amounts of water. Repeat as many times as necessary.

Equipment: container (30-50 gallons)
or
high-pressure spray unit
water
2-3 long-handle, soft-bristle scrub brushes

Station 4: Tape Removal

Remove tape around boots and gloves and deposit in container with plastic liner.

Equipment: container (20-30 gallons)
plastic liners

Station 5: Boot Cover Removal

Remove boot covers and deposit in container with plastic liner.

Equipment: container (30-50 gallons)
plastic liners
bench or stool

Station 6: Outer Glove Removal

Remove outer gloves and deposit in container with plastic liner.

Equipment: container (20-30 gallons)
plastic liners

Station 7: Suit/Safety Boot Wash

Thoroughly wash fully encapsulating suit and boots. Scrub suit and boots with long-handle, soft-bristle scrub brush and copious amounts of decon solution or detergent/water. Repeat as many times as necessary.

Equipment: container (30-50 gallons)
decon solution
or
detergent/water
2-3 long-handle, soft-bristle scrub brushes

Station 8: Suit/Safety Boot Rinse

Rinse off decon solution or detergent/water using copious amounts of water. Repeat as many times as necessary.

Equipment: container (30-50 gallons)
or
high-pressure spray unit
water
2-3 long-handle, soft-bristle scrub brushes

Station 9: Tank Change

If worker leaves Exclusion Zone to change air tank, this is the last step in the decontamination procedure. Worker's air tank is exchanged, new outer gloves and boots covers donned, and joints taped. Worker then returns to duty.

Equipment: air tanks
tape
boot covers
gloves

Station 10: Safety Boot Removal

Remove safety boots and deposit in container with plastic liner.

Equipment: container (30-50 gallons)
plastic liners
bench or stool
boot jack

Station 11: Fully Encapsulating Suit and Hard Hat Removal

With assistance of helper, remove fully encapsulating suit (and hard hat). Hang suits on rack or lay out on drop cloths.

Equipment: rack
drop cloths
bench or stool

Station 12: SCBA Backpack Removal

While still wearing facepiece, remove backpack and place on table. Disconnect hose from regulator valve and proceed to next station.

Equipment: table

Station 13: Inner Glove Wash

Wash with decon solution or detergent/water that will not harm skin. Repeat as many times as necessary.

Equipment: basin or bucket
decon solution
or
detegent/water
small table

Station 14: Inner Glove Rinse

Rinse with water. Repeat as many times as necessary.

Equipment: water
basin or bucket
small table

Station 15: Facepiece Removal

Remove facepiece. Deposit in container with plastic liner. Avoid touching face with fingers.

Equipment: container (30-50 gallons)
plastic liners

Station 16: Inner Glove Removal

Remove inner gloves and deposit in container with plastic liner.

Equipment: container (20-30 gallons)
plastic liners

Station 17: Inner Clothing Removal

Remove clothing soaked with perspiration. Place in container with plastic liner. Do not wear inner clothing off-site since there is a possibility that small amounts of contaminants might have been transferred in removing fully encapsulating suit.

Equipment: container (30-50 gallons)
plastic liners

Station 18: Field Wash

Shower if highly toxic, skin-corrosive or skin-absorbable materials are known or suspected to be present. Wash hands and face if shower is not available.

Equipment: water
soap
small table
basin or bucket
field showers
towels

Station 19: Redress

Put on clean clothes. A dressing trailer is needed in inclement weather.

Equipment: tables
chairs
lockers
clothes

C. FULL DECONTAMINATION (SIT. 1) AND THREE MODIFICATIONS

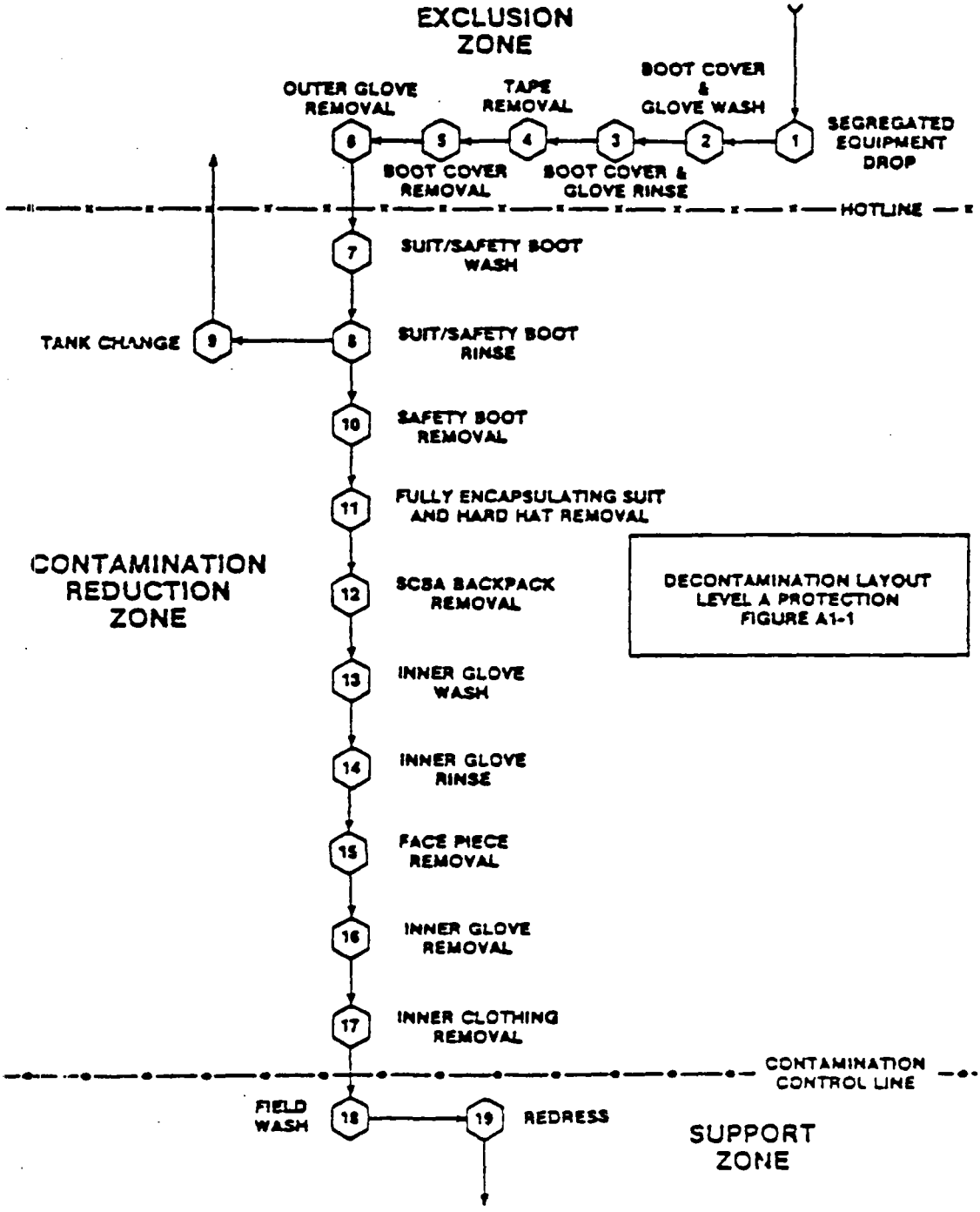
SIT	STATION NUMBER																		
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19
1	X	X	X	X	X	X	X	X		X	X	X	X	X	X	X	X	X	X
2	X	X	X	X	X	X	X	X	X										
3	X						X	X		X	X	X			X	X	X	X	
4	X						X	X	X										

Situation 1: The individual entering the Contamination Reduction Corridor is observed to be grossly contaminated or extremely toxic substances are known or suspected to be present.

Situation 2: Same as Situation 1 except individual needs new air tank and will return to Exclusion Zone.

Situation 3: Individual entering the CRC is expected to be minimally contaminated. Extremely toxic or skin-corrosive materials are not present. No outer gloves or boot covers are worn. Inner gloves are not contaminated.

Situation 4: Same as Situation 3 except individual needs new air tank and will return to Exclusion Zone.



DECONTAMINATION LAYOUT
LEVEL A PROTECTION
FIGURE A1-1

ANNEX 2

LEVEL B DECONTAMINATION

A. EQUIPMENT WORN

The full decontamination procedure outlined is for workers wearing Level B protection (with taped joints between gloves, boot, and suit) consisting of:

- One-piece, hooded, chemical-resistant splash suit.
- Self-contained breathing apparatus.
- Hard hat.
- Chemical-resistant, steel toe and shank boots.
- Boot covers
- Inner and outer gloves.

B. PROCEDURE FOR FULL DECONTAMINATION

Station 1: Segregated Equipment Drop

Deposit equipment used on-site (tools, sampling devices and containers monitoring instruments, radios, clipboards, etc.) on plastic drop cloths or in different containers with plastic liners. Each will be contaminated to a different degree. Segregation at the drop reduces the probability of cross-contamination.

Equipment: various size containers
plastic liners
plastic drop cloths

Station 2: Boot Cover and Glove Wash

Scrub outer boot covers and gloves with decon solution or detergent/ water.

Equipment: container (20-30 gallons)
decon solution
or
detergent water
2-3 long-handle, soft-bristle scrub brushes

Station 3: Boot Cover and Glove Rinse

Rinse off decon solution from Station 2 using copious amounts of water. Repeat as many times as necessary.

Equipment: container (30-50 gallons)
or
high-pressure spray unit
water
2-3 long-handle, soft-bristle scrub brushes

Station 4: Tape Removal

Remove tape around boots and gloves and deposit in container with plastic liner.

Equipment: container (20-30 gallons)
plastic liners

Station 5: Boot Cover Removal

Remove boot covers and deposit in container with plastic liner.

Equipment: container (30-50 gallons)
plastic liners
bench or stool

Station 6: Outer Glove Removal

Remove outer gloves and deposit in container with plastic liner.

Equipment: container (20-30 gallons)
plastic liners

Station 7: Suit/Safety Boot Wash

Thoroughly wash chemical-resistant splash suit, SCBA, gloves, and safety boots. Scrub with long-handle, soft-bristle scrub brush and copious amounts of decon solution or detergent/water. Wrap SCBA regulator (if belt-mounted type) with plastic to keep out water. Wash backpack assembly with sponges or cloths.

Equipment: container (30-50 gallons)
decon solution
or
detergent/water
2-3 long-handle, soft-bristle scrub brushes
small buckets
sponges or cloths

Station 8: Suit/SCBA/Boot/Glove Rinse

Rinse off decon solution or detergent/water using copious amounts of water. Repeat as many times as necessary.

Equipment: container (30-50 gallons)
or
high-pressure spray unit
water
small buckets
2-3 long-handle, soft-bristle scrub brushes
sponges or cloths

Station 9: Tank Change

If worker leaves Exclusion Zone to change air tank, this is the last step in the decontamination procedure. Worker's air tank is exchanged, new outer gloves and boots covers donned, and joints taped. Worker returns to duty.

Equipment: air tanks
tape
boot covers
gloves

Station 10: Safety Boot Removal

Remove safety boots and deposit in container with plastic liner.

Equipment: container (30-50 gallons)
plastic liners
bench or stool
boot jack

Station 11: SCBA Backpack Removal

While still wearing facepiece, remove backpack and place on table. Disconnect hose from regulator valve and proceed to next station.

Equipment: table

Station 12: Splash Suit Removal

With assistance of helper, remove splash suit. Deposit in container with plastic liner.

Equipment: container (30-50 gallons)
plastic liners
bench or stool

Station 13: Inner Glove Wash

Wash inner gloves with decon solution or detergent/water that will not harm skin. Repeat as many times as necessary.

Equipment: decon solution
or
detergent/water
basin or bucket
small table

Station 14: Inner Glove Rinse

Rinse inner gloves with water. Repeat as many times as necessary.

Equipment: water
basin or bucket
small table

Station 15: Facepiece Removal

Remove facepiece. Avoid touching face with gloves. Deposit in container with plastic liner.

Equipment: container (30-50 gallons)
plastic liners

Station 16: Inner Glove Removal

Remove inner gloves and deposit in container with plastic liner.

Equipment: container (20-30 gallons)
plastic liners

Station 17: Inner Clothing Removal

Remove clothing soaked with perspiration. Place in container with plastic liner. Do not wear inner clothing off-site since there is a possibility small amounts of contaminants might have been transferred in removing fully encapsulating suit.

Equipment: container (30-50 gallons)
plastic liners

Station 18: Field Wash

Shower if highly toxic, skin-corrosive, or skin-absorbable materials are known or suspected to be present. Wash hands and face if shower is not available.

Equipment: water
soap

small tables
basins or buckets
field showers

Station 19: Redress

Put on clean clothes. A dressing trailer is needed in inclement weather.

Equipment: tables
chairs
lockers
clothes

C. FULL DECONTAMINATION (SIT. 1) AND THREE MODIFICATIONS

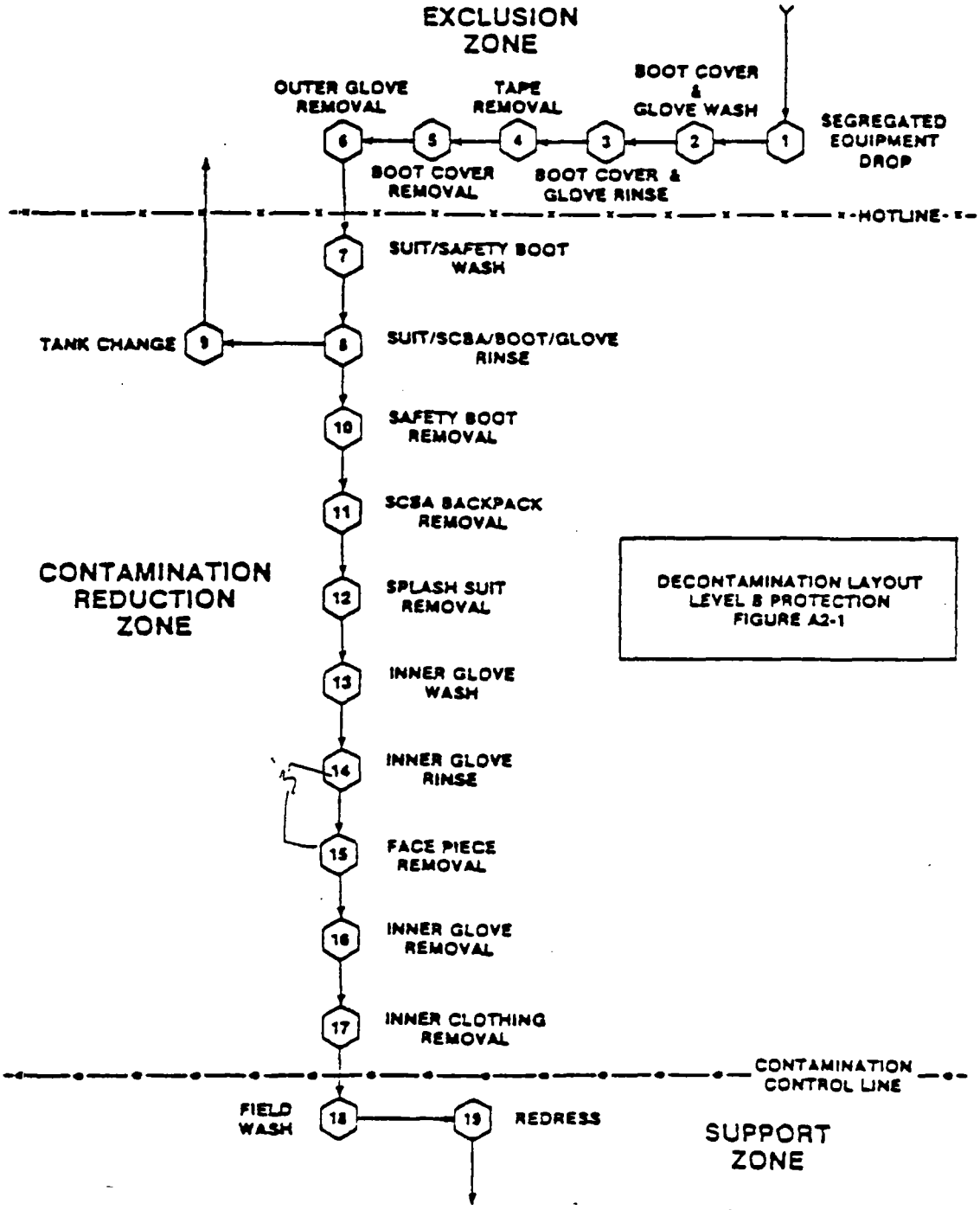
SIT	STATION NUMBER																		
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19
1	X	X	X	X	X	X	X	X		X	X	X	X	X	X	X	X	X	X
2	X	X	X	X	X	X	X	X	X										
3	X						X	X		X	X	X			X	X	X	X	
4	X						X	X	X										

Situation 1: The individual entering the Contamination Reduction Corridor is observed to be grossly contaminated or extremely toxic substances are known or suspected to be present.

Situation 2: Same as Situation 1 except individual needs new air tank and will return to Exclusion Zone.

Situation 3: Individual entering the CRC is expected to be minimally contaminated. Extremely toxic or skin-corrosive materials are not present. No outer gloves or boot covers are worn. Inner gloves are not contaminated.

Situation 4: Same as Situation 3 except individual needs new air tank and will return to Exclusion Zone.



DECONTAMINATION LAYOUT
LEVEL B PROTECTION
FIGURE A2-1

ANNEX 3

LEVEL C DECONTAMINATION

A. EQUIPMENT WORN

The full decontamination procedure outlined is for workers wearing Level C protection (with taped joints between gloves, boots, and suit) consisting of:

- One-piece, hooded, chemical-resistant splash suit.
- Canister equipped, full-face mask.
- Hard hat.
- Chemical-resistant, steel toe and shank boots.
- Boot covers.
- Inner and outer gloves.

B. PROCEDURE FOR FULL DECONTAMINATION

Station 1: Segregated Equipment Drop

Deposit equipment used on-site (tools, sampling devices and containers, monitoring instruments, radios, clipboards, etc.) on plastic drop cloths or in different containers with plastic liners. Each will be contaminated to a different degree. Segregation at the drop reduces the probability of cross-contamination.

Equipment: various size containers
plastic liners
plastic drop cloths

Station 2: Boot Cover and Glove Wash

Scrub outer boot covers and gloves with decon solution or detergent/ water.

Equipment: container (20-30 gallons)
decon solution
or
detergent water
2-3 long-handle, soft-bristle scrub brushes

Station 3: Boot Cover and Glove Rinse

Rinse off decon solution from Station 2 using copious amounts of water. Repeat as many times as necessary.

Equipment: container (30-50 gallons)
or
high-pressure spray unit
water
2-3 long-handle, soft-bristle scrub brushes

Station 4: Tape Removal

Remove tape around boots and gloves and deposit in container with plastic liner.

Equipment: container (20-30 gallons)
plastic liners

Station 5: Boot Cover Removal

Remove boot covers and deposit in container with plastic liner.

Equipment: container (30-50 gallons)
plastic liners
bench or stool

Station 6: Outer Glove Removal

Remove outer gloves and deposit in container with plastic liner.

Equipment: container (20-30 gallons)
plastic liners

Station 7: Suit/Safety Boot Wash

Thoroughly wash splash suit and safety boots. Scrub with long-handle, soft-bristle scrub brush and copious amounts of decon solution or detergent/water. Repeat as many times as necessary.

Equipment: container (30-50 gallons)
decon solution
or
detergent/water
2-3 long-handle, soft-bristle scrub brushes

Station 8: Suit/Safety Boot Rinse

Rinse off decon solution or detergent/water using copious amounts of water. Repeat as many times as necessary.

Equipment: container (30-50 gallons)
or
high-pressure spray unit
water
2-3 long-handle, soft-bristle scrub brushes

Station 9: Canister or Mask Change

If worker leaves Exclusion Zone to change canister (or mask), this is the last step in the decontamination procedure. Worker's canister is exchanged, new outer gloves and boots covers donned, and joints taped. Worker returns to duty.

Equipment: canister (or mask)
tape
boot covers
gloves

Station 10: Safety Boot Removal

Remove safety boots and deposit in container with plastic liner.

Equipment: container (30-50 gallons)
plastic liners
bench or stool
boot jack

Station 11: Splash Suit Removal

With assistance of helper, remove splash suit. Deposit in container with plastic liner.

Equipment: container (30-50 gallons)
bench or stool
plastic liner

Station 12: Inner Glove Wash

Wash inner gloves with decon solution or detergent/water that will not harm skin. Repeat as many times as necessary.

Equipment: decon solution
or
detergent/water
basin or bucket

Station 13: Inner Glove Rinse

Rinse inner gloves with water. Repeat as many times as necessary.

Equipment: water
basin or bucket
small table

Station 14: Facepiece Removal

Remove facepiece. Avoid touching face with gloves. Deposit facepiece in container with plastic liner.

Equipment: container (30-50 gallons)
plastic liners

Station 15: Inner Glove Removal

Remove inner gloves and deposit in container with plastic liner.

Equipment: container (20-30 gallons)
plastic liners

Station 16: Inner Clothing Removal

Remove clothing soaked with perspiration. Place in container with plastic liner. Do not wear inner clothing off-site since there is a possibility small amounts of contaminants might have been transferred in removing fully encapsulating suit.

Equipment: container (30-50 gallons)
plastic liners

Station 17: Field Wash

Shower if highly toxic, skin-corrosive or skin-absorbable materials are known or suspected to be present. Wash hands and face if shower is not available.

Equipment: water
soap
tables
wash basins/buckets
field showers

Station 18: Redress

Put on clean clothes. A dressing trailer is needed in inclement weather.

Equipment: tables
chairs
lockers
clothes

C. FULL DECONTAMINATION (SIT. 1) AND THREE MODIFICATIONS

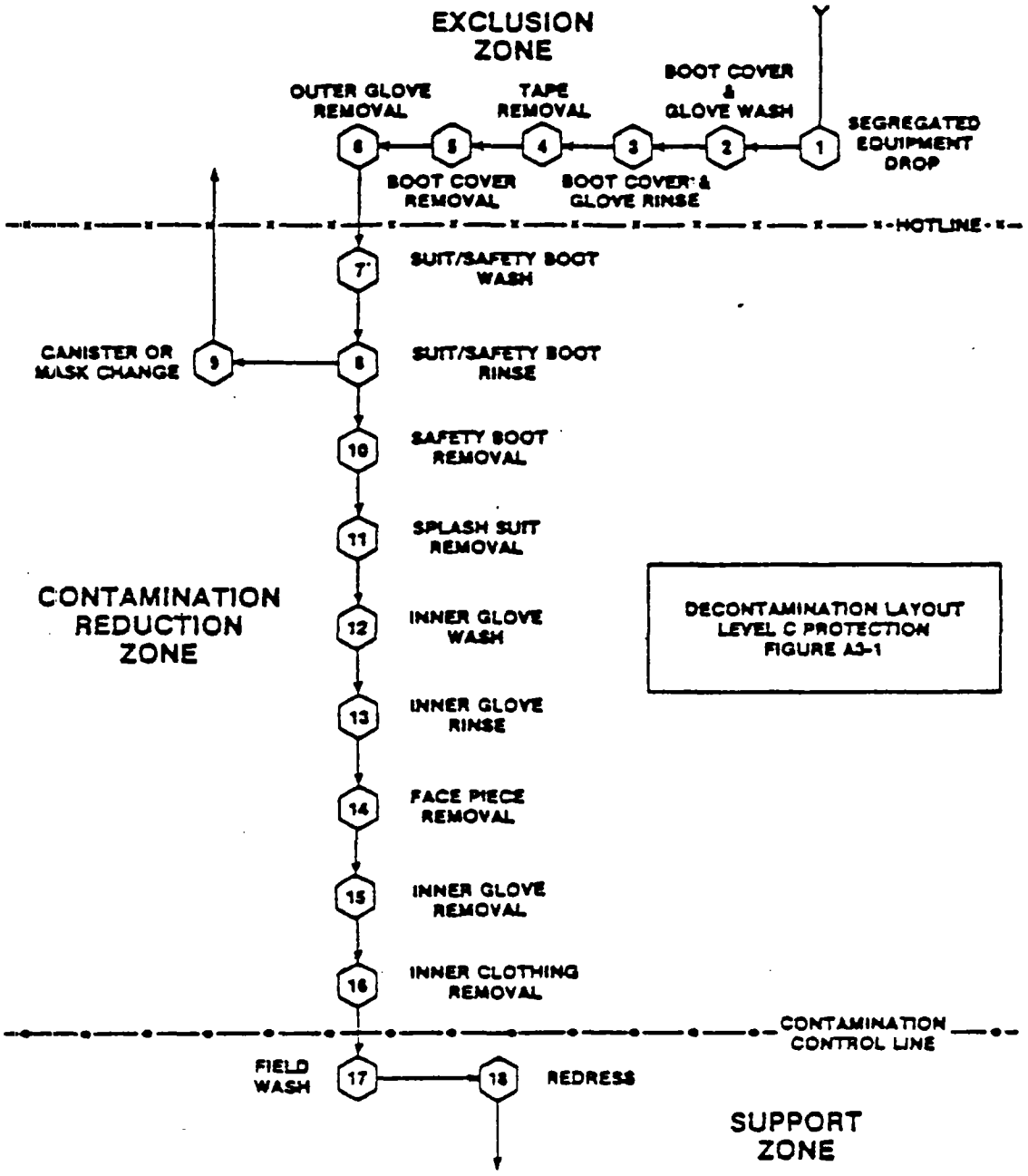
SIT	STATION NUMBER																	
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1	X	X	X	X	X	X	X	X		X	X	X	X	X	X	X	X	X
2	X	X	X	X	X	X	X	X	X									
3	X						X	X		X	X			X	X	X	X	
4	X						X	X	X									

Situation 1: The individual entering the Contamination Reduction Corridor is observed to be grossly contaminated or extremely skin-corrosive substances are known or suspected to be present.

Situation 2: Same as Situation 1 except individual needs new canister or mask and will return to Exclusion Zone.

Situation 3: Individual entering the CRC is expected to be minimally contaminated. Extremely skin-corrosive materials are not present. No outer gloves or boot covers are worn. Inner gloves are not contaminated.

Situation 4: Same as Situation 3 except individual needs new canister or mask and will return to Exclusion Zone.



DECONTAMINATION LAYOUT
LEVEL C PROTECTION
FIGURE A3-1

ANNEX 4

LEVEL A DECONTAMINATION, MINIMUM LAYOUT

A. EQUIPMENT WORN

The decontamination procedure outlined is for workers wearing Level A protection (with taped joints between gloves, boots, and suit) consisting of:

- Fully encapsulating suit with integral boots and gloves.
- Self-contained breathing apparatus.
- Hard hat (optional).
- Chemical-resistant, steel toe and shank boots.
- Boot covers.
- Inner and outer gloves.

B. PROCEDURE FOR FULL DECONTAMINATION

Station 1: Segregated Equipment Drop

Deposit equipment used on-site (tools, sampling devices and containers, monitoring instruments, radios, clipboards, etc.) on plastic drop cloths or in different containers with plastic liners. Each will be contaminated to a different degree. Segregation at the drop reduces the probability of cross-contamination.

Equipment: various size containers
plastic liners
plastic drop clothes

Station 2: Outer Garment, Boots, and Gloves Wash and Rinse

Scrub outer boots, outer gloves, and fully-encapsulating suit with decon solution or detergent water. Rinse off using copious amounts of water.

Equipment: containers (30-50 gallons)
decon solution
or
detergent water
rinse water
2-3 long-handle, soft-bristle scrub brushes

Station 3: Outer Boot and Glove Removal

Remove outer boots and gloves. Deposit in container with plastic liner.

Equipment: container (30-50 gallons)
plastic liners
bench or stool

Station 4: Tank Change

If worker leaves Exclusion Zone to change air tank, this is the last step in the decontamination procedure. Worker's air tank is exchanged, new outer gloves and boot covers donned, joints taped, and worker returns to duty.

Equipment: air tanks
tape
boot covers
gloves

Station 5: Boot, Gloves, and Outer Garment Removal

Boots, fully-encapsulating suit, and inner gloves removed and deposited in separate containers lined with plastic.

Equipment: containers (30-50 gallons)
plastic liners
bench or stool

Station 6: SCBA Removal

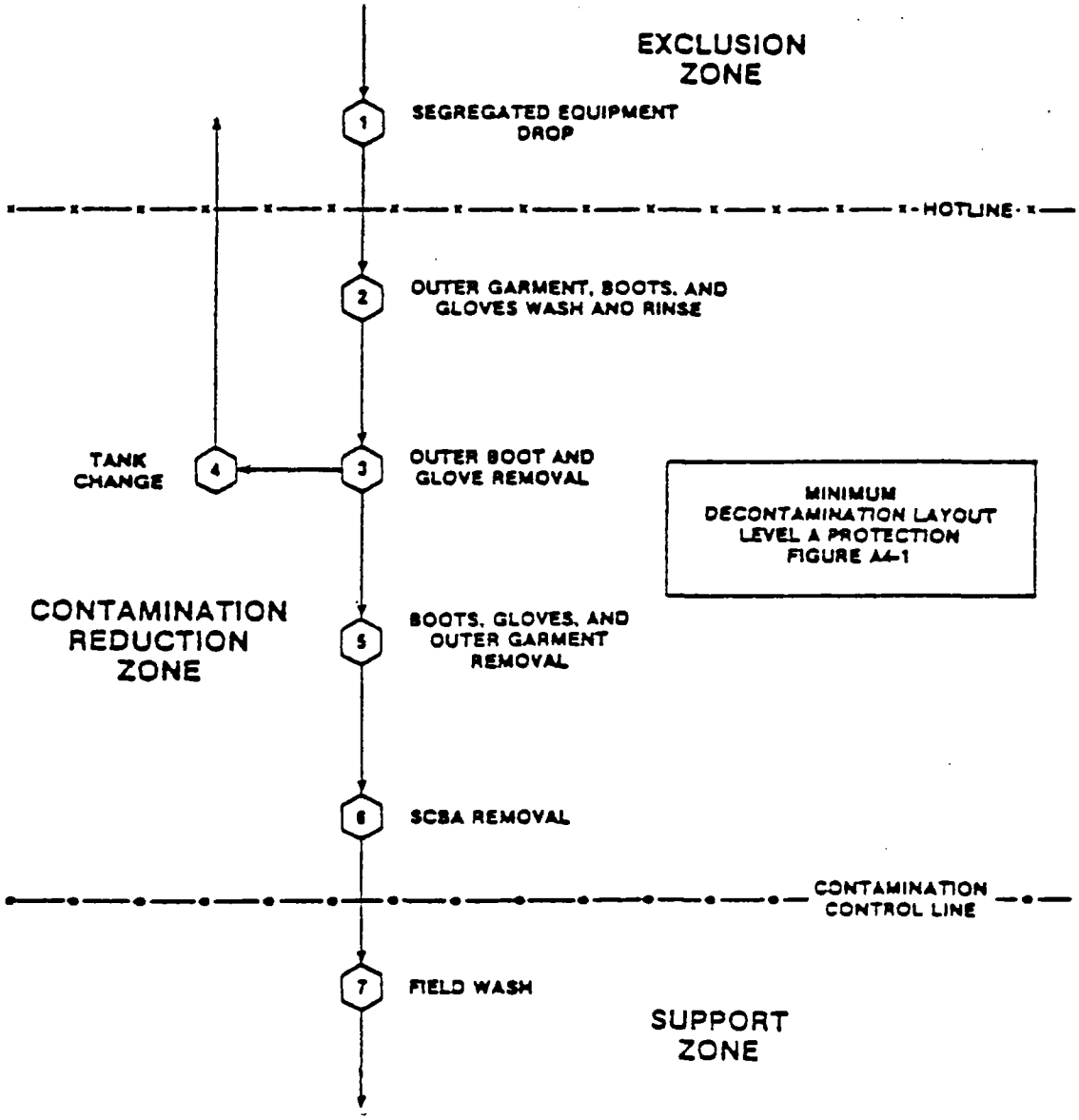
SCBA backpack and facepiece is removed. Hands and face are thoroughly washed. SCBA deposited on plastic sheets.

Equipment: plastic sheets
basin or bucket
soap and towels
bench

Station 7: Field Wash

Thoroughly wash hands and face. Shower as soon as possible.

Equipment: water
soap
tables
wash basin/bucket



MINIMUM DECONTAMINATION LAYOUT LEVEL A PROTECTION FIGURE A4-1